

OPERATING INSTRUCTIONS

DIBORANE SHIPPING CONTAINER

D.O.T. SPECIAL PERMIT NO. 6522

DANGER

DIBORANE: Compressed gas, n.o.s. (Flammable)

Poisonous gas, n.o.s.

IMPORTANT WARNING

If this container is not delivered within
the specified shipping time, Carrier must:

Call BUREAU OF EXPLOSIVES (202) 293-4084

**CASE FILE
COPY**

MANUAL PUBLISHED JUNE 1972

by

CALLERY CHEMICAL COMPANY
DIVISION OF MINE SAFETY APPLIANCES COMPANY
CALLERY, PENNSYLVANIA
16024

Revision 1; September, 1972

FOREWORD

This manual of Operating Instructions was prepared under Contract Number NASW 1827; sponsored by the National Aeronautics and Space Administration, Jet Propulsion Laboratory. The work was done under the technical direction of Walter B. Powell of the Jet Propulsion Laboratory in Pasadena California.

DIBORANE HANDBOOK, prepared for NASA by Rocketdyne (Division of North American Rockwell Corporation) under Contract NAS7-769, is referenced herein for more detailed information. That compilation includes experience of both NASA and Callery (and others), so every effort was made to make this manual compatible with that reference.

NOTICE

The information contained in this manual of operating instructions is based on the experience record compiled by Callery Chemical Company, Division of Mine Safety Appliances Company; and is offered to diborane users as a guide for correct and safe operating procedures with the 200-pound shipping container.

To some extent these procedures must be written in general terms because of differences between the facilities of those receiving the diborane. Furthermore, in terms of shipping regulations, an attempt was made to point out those regulations most pertinent to the 200-pound diborane shipments made under Department of Transportation Special Permit Number 6522; however, the responsibility of compliance with all applicable Department of Transportation regulations remains as always with the shipper.

Although Callery Chemical Company believes the information in this operating manual is accurate and, if the instructions are followed, will result in safe and efficient operations, Callery Chemical Company cannot and does not assume responsibility either for the accuracy of the information or for any operation not under the direct supervision of Callery personnel.

One purpose of this operating manual is to inform the end user of the potential hazards that may be encountered, and to present some basic precautions to be followed to prevent needless accidents. It is the customer's responsibility to insure that this manual is read by both the supervisors and by the men actually working with the products. The ultimate responsibility of ensuring that safe practices are followed is with the management and supervision of the companies using the material.

DIBORANE SHIPPING CONTAINER
OPERATING INSTRUCTIONS

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INTRODUCTION

The purpose of this manual is to provide instructions and information for safe and efficient use of the 200-pound diborane shipping container. Scope of the manual is to include: Shipping, Receiving, Storage, Unloading, Return, Cleanout, and Loading. Backup information on container design and performance, diborane information, etc. are also included. It is strongly recommended that the container design section with drawings be carefully reviewed before attempting to follow the procedures.

To assure that this manual reaches those persons who need the information, the following procedure is recommended for dissemination:

1. One copy should be sent in advance of any shipment, to the individual designated by consignee as responsible for receiving, storing, and unloading the container. This will enable the user to make preparations in advance.
2. One copy should accompany the shipment, fastened to the container so that it remains available to the person working with the container. This copy would contain data sheets for entry of necessary information on each activity, and would remain with the container.

3. Another copy could also be sent inside the valve compartment of the container to be removed for the convenience of others who need the information.

This manual includes complete information only on the shipping container itself, and cannot cover the complete requirements of whatever facility might be used for diborane loading and unloading. The users of diborane are cautioned to make sure their transfer and storage facilities are properly designed, built, and maintained.

I DESIGN AND PERFORMANCE

DESIGN

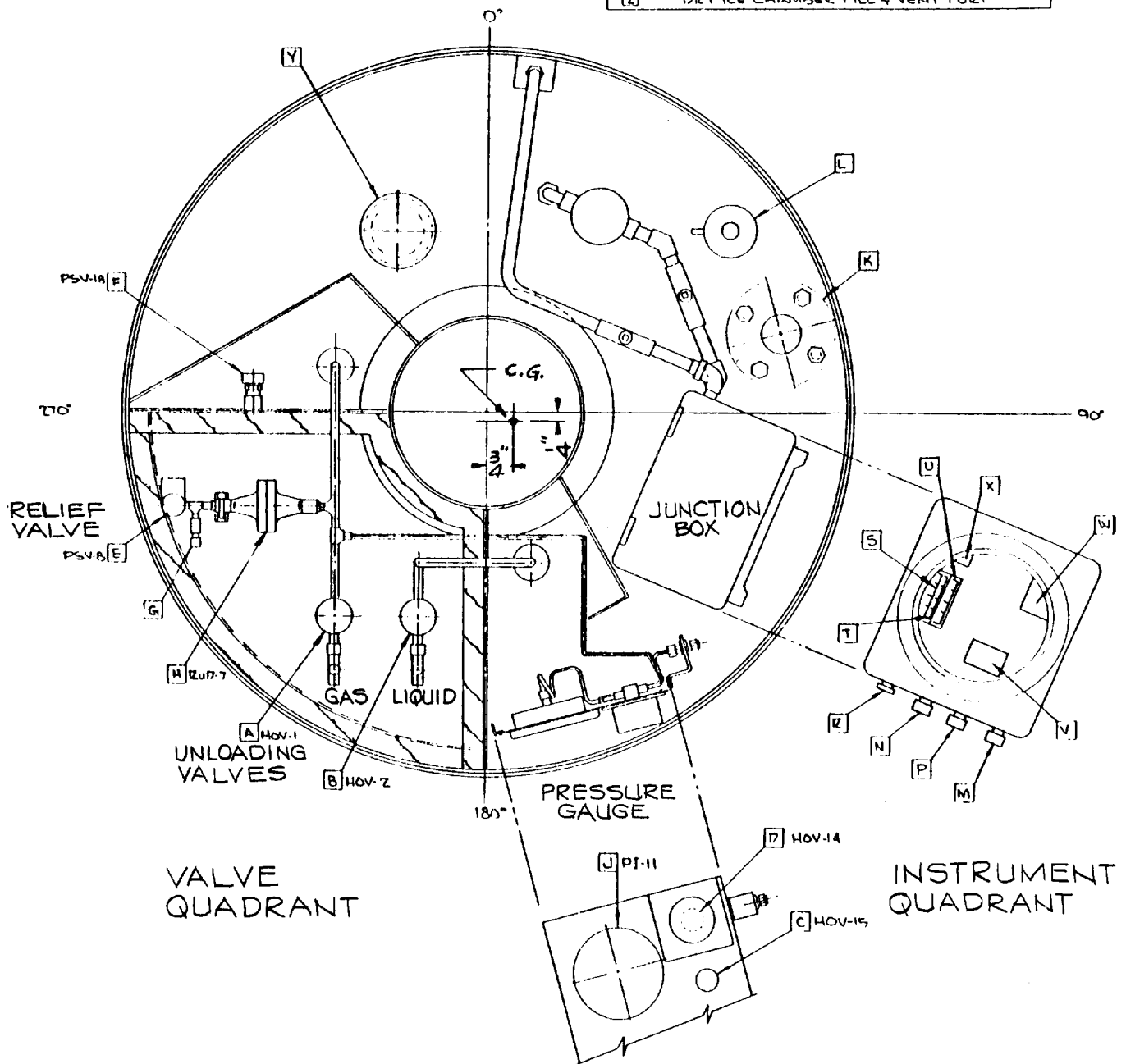
A. General Design

Design concept for this container was selected to take full advantage of the success achieved in shipping diborane for over fifteen years. On this basis, dry ice was chosen as the refrigerant; because this eliminates the need for control devices used in liquid nitrogen or other liquid flow-type refrigeration systems. In addition dry ice is comparatively much easier to obtain and add to a container which has been delayed in transit.

Reference to Figure 1 and Figure 2 will show the general layout of this container; with a 36-inch diameter spherical inner tank for the diborane, surrounded (except for the dry ice chamber) by a 48-inch diameter cylindrical shell containing perlite insulation evacuated to about ten microns absolute pressure.

FIGURE 2
DIBORANE SHIPPING CONTAINER
PLAN VIEW

ITEM	DESCRIPTION
A	VENT VALVE & PRESSURIZATION VALVE
B	FILL VALVE & LIQUID UNLOADING VALVE
C	PRESSURE GAGE ISOLATION VALVE
D	PRESSURE GAGE PURGE VALVE
E	SAFETY VALVE SET @ 550 PSIG
F	SAFETY VALVE SET @ .5 PSI
G	EXCESS FLOW VALVE
H	RUPTURE DISC SET @ 550 PSIG
I	PRESS. GAGE -30" VAC -0 TO 1000 PSIG RANGE
J	FILTER ASSY W/ WASTINGS DVGW VAC GAGE
K	LINDE OPERATOR F/2" PUMP OUT VALVE
L	TEST-PUSH BUTTON
M	SILENCE-PUSH BUTTON
N	LEVEL INDICATOR PUSH BUTTON
O	DRY ICE CHAMBER
P	ON-OFF SELECTOR SWITCH
Q	VOLTMETER-LINEAR SCALE
R	LIQUID LEVEL INDICATOR
S	TEMPERATURE INDICATOR (-130° C. TO +130° C.)
T	CONTROL RELAY - 24V
U	BATTERY - 27V, 3,400 MAH
V	VISUAL & AUDIO ALARM
W	PERLITE FILL PORT
X	DRY ICE CHAMBER FILL & VENT PORT



Over 100 pounds of dry ice may be added through a bellows-sealed neck into a chamber in direct contact with the top of the sphere. The spherical tank containing the diborane is coded for a maximum working pressure of 500 psig at -320 to +100°F., protected by a 550 psig rupture disk and 550 psig relief valve connected in series. An excess flow valve prevents pressure buildup between the rupture disk and relief valve.

In keeping with end use requirements of NASA, the liquid unloading rate was designed for a minimum of 0.2 pound per second with 100 psi pressure differential. Actual unloading rate of 0.2 pound per second was obtained with a pressure differential of only 20 psi.

The container is designed for handling by fork lift or by crane; gross shipping weight is about 2900 pounds. Envelope dimensions are 4'-4" diameter and 6'-6" height. Center of gravity is shown on Figure 1 and Figure 2.

B. Specifications

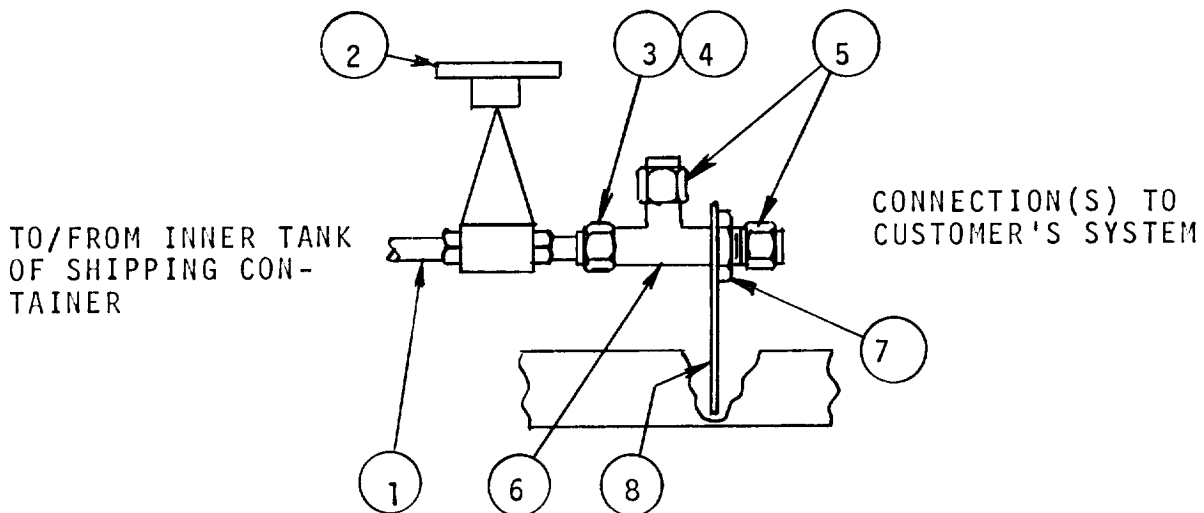
A summary of user specifications for the diborane shipping container is given in Table 1, along with Figure 3 -- the two pages following.

TABLE 1
DIBORANE SHIPPING CONTAINER
SPECIFICATIONS

DOT Special Permit	No. 6522
Capacity:	
Inner Tank Volume	14.14 ft. ³ = 24,400 in. ³ = 105.7 gal.
Diborane Weight	100 lbs. minimum
	200 lbs. maximum
Dry Ice Weight	over 108 pounds
Service Pressure:	
Maximum Working	500 psig @ -320 to +100°F.
Recommended Operating	400 psig maximum
Gross Shipping Weight	Approx. 2900 pounds
Tare Weight	Approx. 2590 pounds
*Liquid Connection (Dip Tube)	1/2" OD Tubing Tee, AN804C8 (horizontal run) Downstream of 1/2" Bellows Valve, Nupro SS-8BG-Sw
*Gas Connection (Top of Tank)	1/2" OD Tubing Tee, AN804C8 (horizontal run) Downstream of 1/2" Bellows Valve, Nupro SS-8BG-Sw

*See Figure 3, Next Page

FIGURE 3
DIAGRAM OF CONNECTIONS FOR UNLOADING DIBORANE
(Same for Liquid and Gas)



1. 1/2" OD X 0.049" Wall Seamless Tubing, Stn. Stl.
2. 1/2" Valve, Bellows Type, Nupro No. SS-8 BG-SW, Stn. Stl.
3. 3/4"-16 UNF-3B Union Nut, AN805C8. Stn. Stl.
4. 1/2" OD Sleeve Coupling, AN819-8C, Stn. Stl.
5. 1/2" OD Cap Assembly, AN929-8C, Stn. Stl.
6. 1/2" OD Tee with Bulkhead on Run, AN804-C8, Stn. Stl.
7. 3/4"-16 UNF-3B Nut, AN924-8C, Stn. Stl.
8. 1/4" X 2" X 5" Plate, Stn. Stl.

C. Instruments and Controls

A schematic diagram showing instruments and controls is given in Figure 4, on the following page.

Details on the location, operation, and specifications of individual instruments and controls are given on the pages following:

<u>Item</u>	<u>Instrument</u>	<u>Figure</u>	<u>Page</u>
PI-11	Pressure Indicator	5	11
TI-12	Temperature Indicator	6	12
LI-13	Level Indicator	7	13
VI	Voltage Indicator	8	16
TA-17	Audio-Visual Alarm	9	17
RuD-7 PSV-8 EFV-19 PSV-18	Safety Relief Devices	10	18

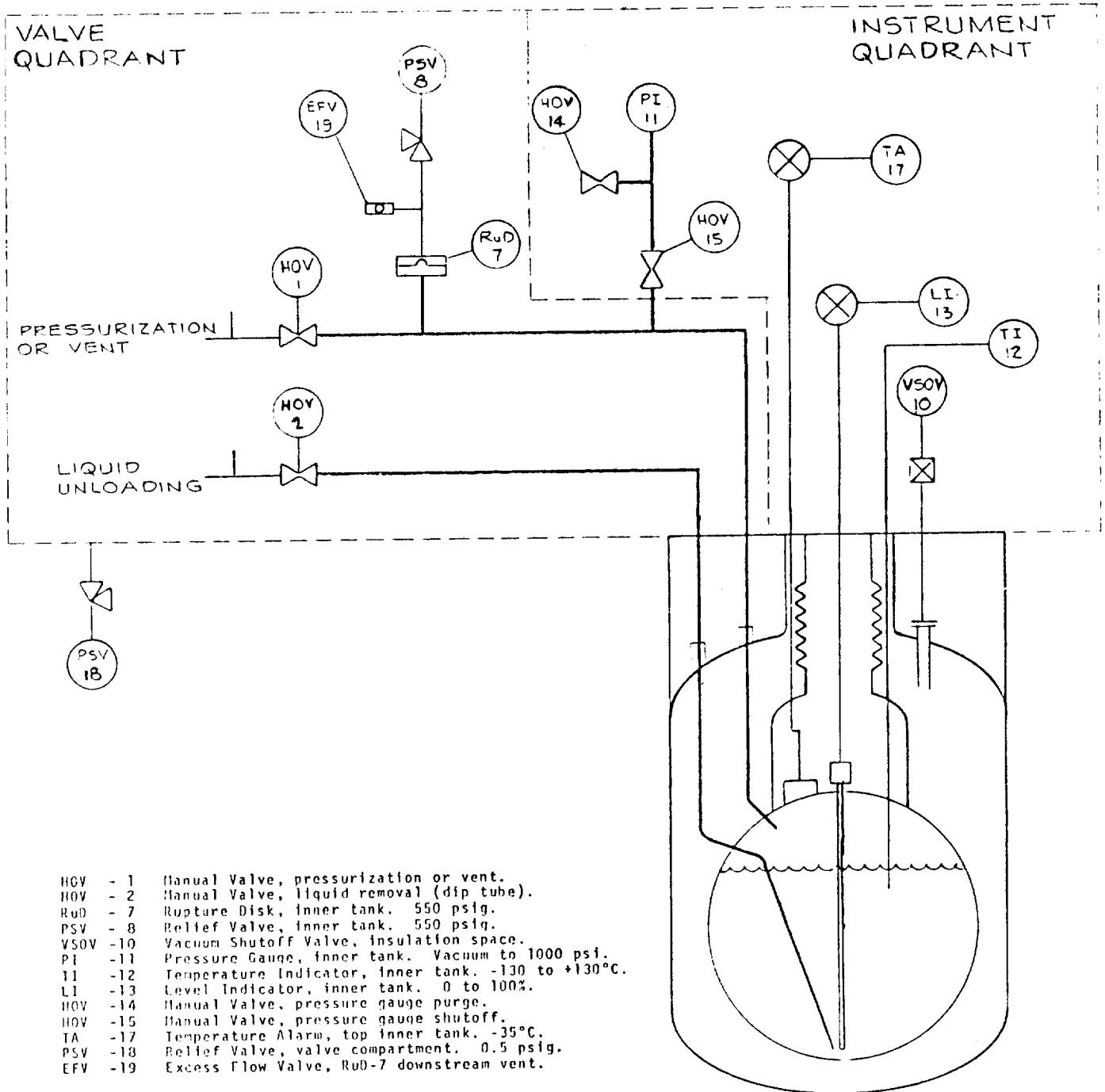
The following drawings show additional details on Piping & Instrumentation and Electrical

Piping & Instrumentation-1	11	19
Piping & Instrumentation-2	12	20
Electrical, Junction Box	13	21
Electrical, Wiring Diagram	14	22

FIGURE 4

- 10 -

SCHEMATIC DIAGRAM CONTAINER INSTRUMENTS AND CONTROLS



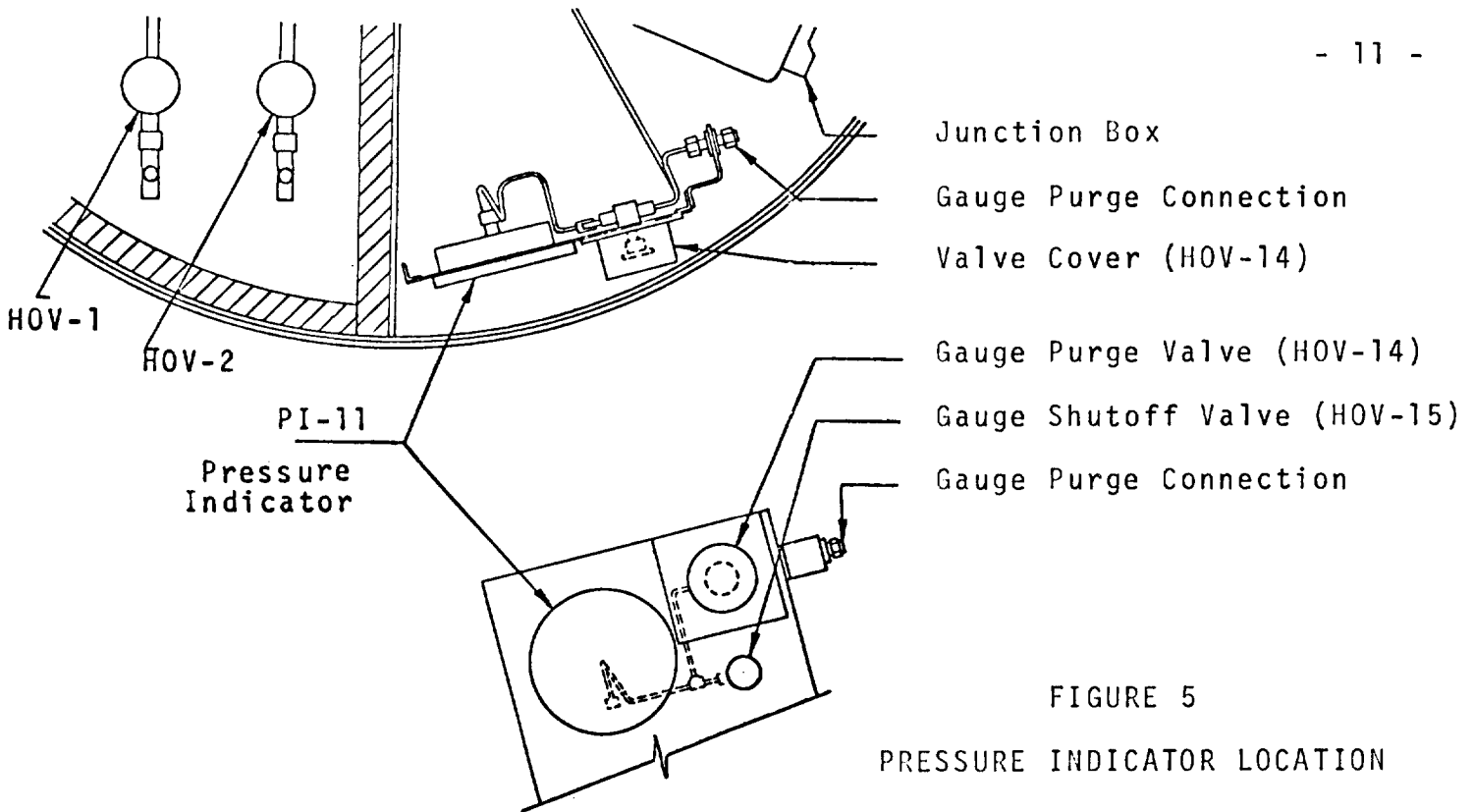


FIGURE 5
PRESSURE INDICATOR LOCATION

1. Pressure is indicated by a pressure gauge (PI-11 in Figure 2, Figure 4, and Figure 5) with range of full vacuum to 1000 psi. The gauge is located behind a plexiglass window in the removable side panel, therefore can be viewed remotely if necessary. Manual valve HOV-15 may be used to isolate the gauge; however, this valve is left open during shipment and should be left open during storage. Another manual valve, HOV-14, permits making connection to purge the line to the gauge.

SPECIFICATIONS:

- | | |
|--------|---|
| PI-11 | Pressure Gauge, 4-1/2" dial, 30" vacuum to 1000 psi range, flush mounting, 1/2" back connected. Ashcroft Maxisafe #1377-SS. |
| HOV-14 | Hand-operated Valve, 1/4" tube socket weld, ends, Nupro #SS-4H-SW. |
| HOV-15 | Hand-operated Valve, 1/4" tube socket weld ends, Nupro #SS-4H-SW. |

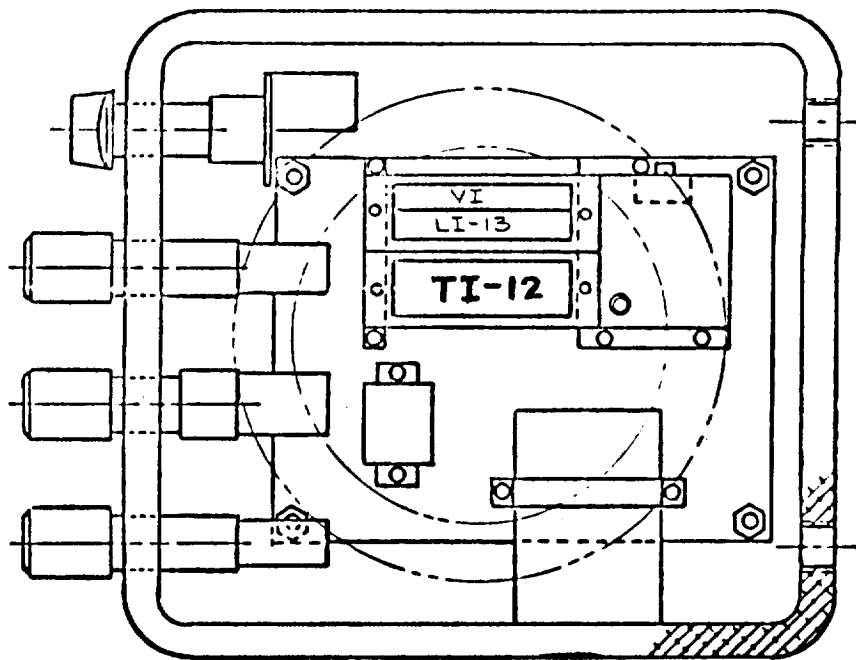


FIGURE 6
TEMPERATURE INDICATOR LOCATION

2. Temperature is given by an indicator (TI-12 in Figure 2, Figure 4, and Figure 6), from a temperature sensing element (thermocouple) at the top of the inner tank (Figure 1). Range is -130°C. to $+130^{\circ}\text{C.}$ The thermocouple, inserted from the dry ice chamber, extends down into the inner tank (Figure 1); but does not reach the liquid, so the indicated temperature is that of the vapor in equilibrium with the liquid. Experience has shown that this temperature reading is influenced by the presence of dry ice in the chamber above. The temperature indicator is continuous reading, requiring no power supply (ie, does not require a battery); and is also visible through a plexiglass window in the side panel.

SPECIFICATIONS (TI-12):

Sensing Element -- Miniature Ceramocouple with 24" Fiberglass leads, #5 mounting bushing, 6" immersion length and short transition. Thermo-Electric #5K1521A.

Indicator -- Indicating Pyrometer. International Instrument #2501-HL-I/C- (-130) - $(+130)^{\circ}\text{C.}$

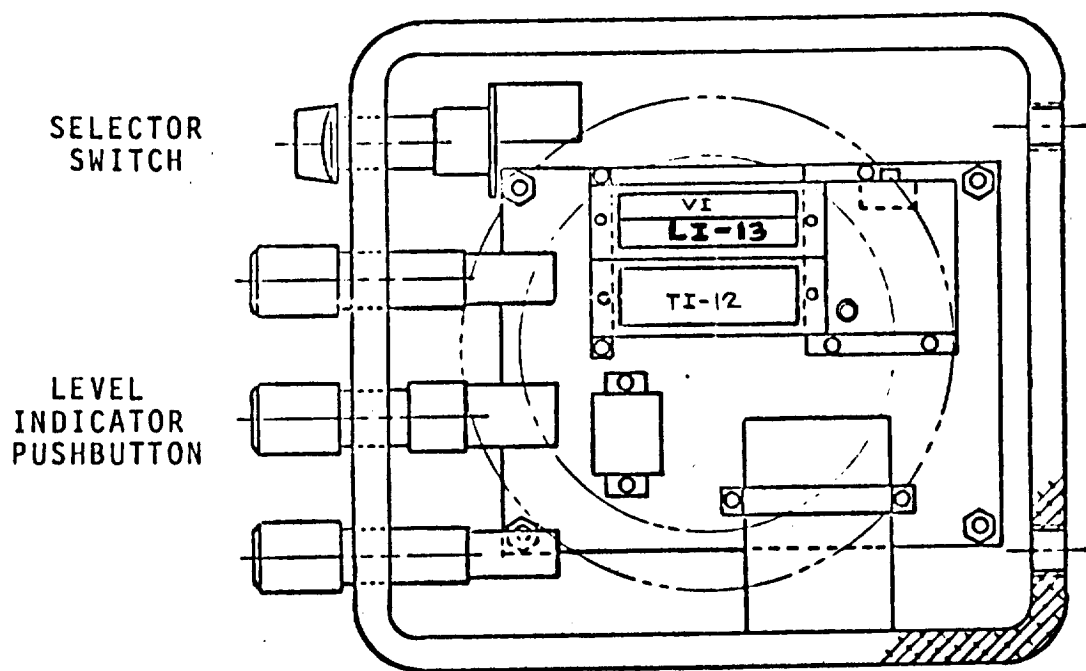


FIGURE 7
LEVEL INDICATOR LOCATION

3. Liquid Level is indicated on a percentage scale just above the temperature scale. The level indicator (LI-13 on Figure 2, Figure 4, and Figure 7) reads only when activated by the controls along the left side of the junction box. The selector switch (top control) must be in the "on" position, then the level indicator may be activated by pushing and holding in the center of three push-buttons below the selector switch (Figure 7). Liquid level is sensed by a capacitance probe which is inserted from the dry ice chamber and extends nearly to the bottom of the inner tank (Figure 1). Conversion of liquid level to weight of diborane requires knowledge of temperature, since diborane density is greatly affected by temperature. Table 2 gives diborane weight versus level indicator reading as a function of temperature. This weight will be only an approximate value. LIQUID LEVEL READING MUST NOT BE USED AS A BASIS FOR DETERMINING WHETHER CONTAINER IS "EMPTY" FOR RETURN (see Procedure for Return, Section X).

SPECIFICATIONS (LI-12)

Indicator -- Dual Movement Meter with interchangeable scale.
International Instrument #2520-HK-5DCMA-5DCMA.

Note: This same indicator is also used as the indicator for voltage, VI. Liquid level reads on the lower scale, and battery voltage reads on the upper scale.

Sensing Element -- Capacitance Probe, 7/16" diameter, concentric shielded probe, high gain, non-insulated for linear measurement and sufficient signal, 3 feet long, 3/4" N.P.T. connection. Robertshaw Controls Company Probe P/N 150-800-075.

Transmitter -- Self-Contained Transmitter Only (indicator not supplied), Robertshaw Level-Tel Transmitter. Robertshaw Controls Company Model 156-A1

The Model 156 transmitter is a capacitance operated DC current transmitter providing output current directly proportional to input capacitance change. Non-interacting and independent zero and span adjustments are provided for calibration.

Input capacitance measurement is accomplished by driving the terminal or probe capacitance with a constant current generator providing a linear voltage ramp across the capacitance whose slope is proportional to the capacitance value. When the ramp voltage reaches a fixed reference voltage, a solid state switch discharges the input capacitance providing an output pulse to a shaping network and pulse integrator. DC output voltage from the integrator is then used as a feedback current to adjust the charging current generator to maintain the ramp period (frequency) at a constant value. The feedback current also drives the DC current output stage providing an output current directly proportional to input capacitance change.

Performance: Linearity, + or - 0.25%
Repeatability, + or - 0.1%
Sensitivity, 0.01% minimum
Frequency Response, -3db at 1 cps
Supply Variation Effect, 0.5%/10% supply
Ambient Temperature Effect
Span, 0.01%/°F.
Zero, 0.01%/°F. or 0.003 Pf/°F.
whichever is greater
Output Signal Ripple, 0.25% maximum
at twice power line frequency.

TABLE 2
DIBORANE SHIPPING CONTAINER
LEVEL INDICATOR CALIBRATION

Level Reading	Total Diborane Weight, Pounds @ Temperature, °C.											
	-78°	-75°	-70°	-65°	-60°	-55°	-50°	-45°	-40°	-35°	-30°	0°
0	3.5	4.1	4.8	6.0	7.0	8.7	7.0	12.2	13.8	16.7	16.7	51.7
2	4.5	5.1	5.8	7.0	7.9	9.6	7.9	13.0	14.7	17.6	17.6	52.3
4	6.3	6.9	7.6	8.7	9.6	11.2	9.6	14.6	16.2	19.1	19.1	53.3
6	8.9	9.4	10.1	11.2	12.1	13.6	11.9	16.9	18.5	21.2	21.2	54.7
8	12.2	12.7	13.3	14.4	15.2	16.7	14.9	19.8	21.3	23.9	23.9	56.5
10	16.3	16.7	17.2	18.2	19.0	20.3	18.6	23.3	24.7	27.2	27.2	58.7
12	21.0	21.3	21.8	22.7	23.4	24.6	22.8	27.4	28.7	31.1	31.1	61.2
14	26.3	26.6	27.0	27.8	28.4	29.5	27.6	32.0	33.3	35.5	35.5	64.1
16	32.2	32.5	32.8	33.4	33.9	34.9	33.0	37.2	38.3	40.4	40.4	67.4
18	38.7	38.9	39.1	39.6	40.0	40.9	38.9	42.8	43.9	45.7	45.7	70.9
20	45.8	45.8	45.9	46.3	46.6	47.3	45.2	48.9	49.8	51.5	51.5	74.7
22	53.3	53.2	53.3	53.4	53.7	54.1	52.1	55.5	56.2	57.7	57.7	78.8
24	61.3	61.1	61.0	61.0	61.1	61.4	59.3	62.4	63.1	64.2	64.2	83.2
26	69.7	69.4	69.2	69.0	69.0	69.1	66.9	69.8	70.2	71.1	71.1	87.8
28	78.5	78.1	77.8	77.4	77.3	77.2	74.9	77.4	77.7	78.4	78.4	92.6
30	87.7	87.2	86.7	86.1	85.8	85.5	83.1	85.4	85.5	85.9	85.9	97.5
32	97.1	96.5	95.9	95.2	94.7	94.2	91.7	93.7	93.6	93.7	93.7	102.7
34	106.9	106.2	105.4	104.5	103.9	103.1	100.6	102.2	101.9	101.7	101.7	108.0
36	116.9	116.1	115.2	114.0	113.3	112.3	109.6	110.9	110.5	110.0	110.0	113.5
38	127.2	126.2	125.2	123.8	122.9	121.6	118.9	119.8	119.2	118.4	118.4	119.1
40	137.6	136.5	135.3	133.7	132.6	131.1	128.3	128.9	128.1	126.9	126.9	124.8
42	148.2	146.9	145.6	143.8	142.5	140.8	137.9	138.1	137.1	135.6	135.6	130.5
44	158.9	157.4	156.0	153.9	152.6	150.6	147.6	147.4	146.2	144.4	144.4	136.3
46	169.7	168.1	166.5	164.2	162.7	160.4	157.3	156.8	155.3	153.3	153.3	142.2
48	180.5	178.7	177.0	174.5	172.8	170.3	167.1	166.2	164.6	162.2	162.2	148.1
50	191.3	189.4	187.6	184.8	182.9	180.2	176.9	175.6	173.8	171.1	171.1	154.0
52	-	-	198.1	195.1	193.0	190.0	186.7	185.0	183.0	179.9	179.9	159.9
54	-	-	-	-	-	199.8	196.4	194.4	192.1	188.8	188.8	165.7
56	-	-	-	-	-	-	-	-	-	197.5	197.5	171.5
58	-	-	-	-	-	-	-	-	-	-	-	177.3
60	-	-	-	-	-	-	-	-	-	-	-	182.9
62	-	-	-	-	-	-	-	-	-	-	-	188.4
64	-	-	-	-	-	-	-	-	-	-	-	193.8
66	-	-	-	-	-	-	-	-	-	-	-	199.1
68	-	-	-	-	-	-	-	-	-	-	-	-

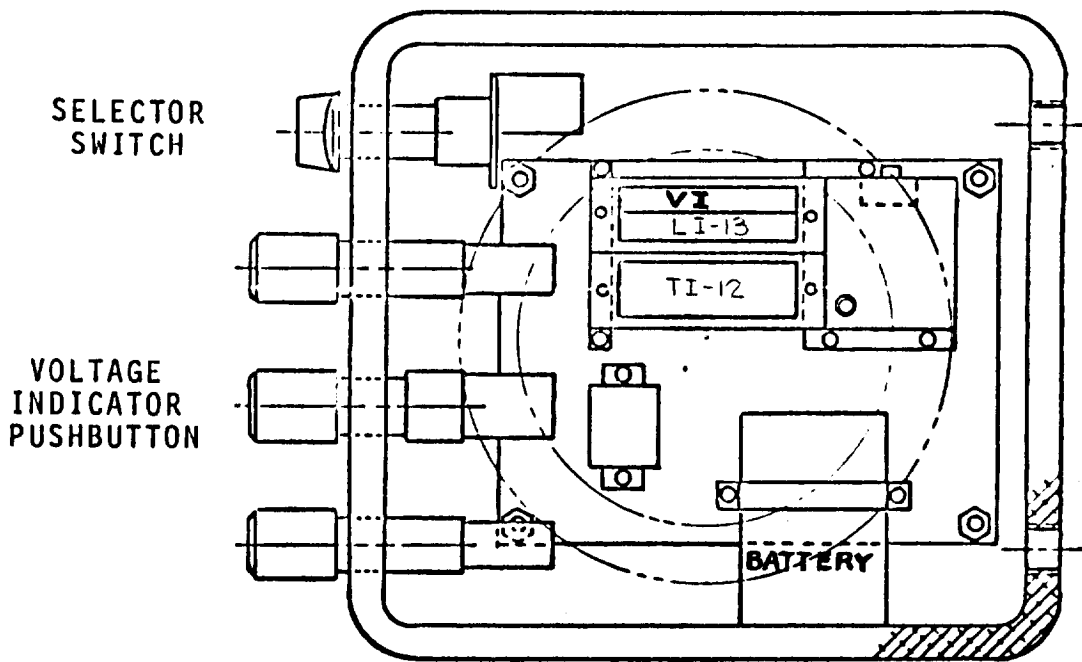


FIGURE 8

4. Voltage is indicated by a separate pointer at the same time as liquid level, on a percentage scale just above the level indicator scale. This voltage shows the condition of the battery which powers the temperature-actuated audio-visual alarm and the level indicator. The voltage indicator (Figure 2 and Figure 8) has a red-lined portion which indicates low battery voltage. This may be due to low ambient temperature, in which case the reading will rise when the container is moved to a higher ambient temperature.

SPECIFICATIONS:

VI-Indicator -- Dual Movement Meter with interchangeable scale.
International Instrument #2520-HK-5DCMA-5DCMA

Note: This same indicator is also used for indicating liquid level on the lower scale, with voltage on the upper scale.

Battery -- Burgess Battery #302580; 27v, 3,400 MAH

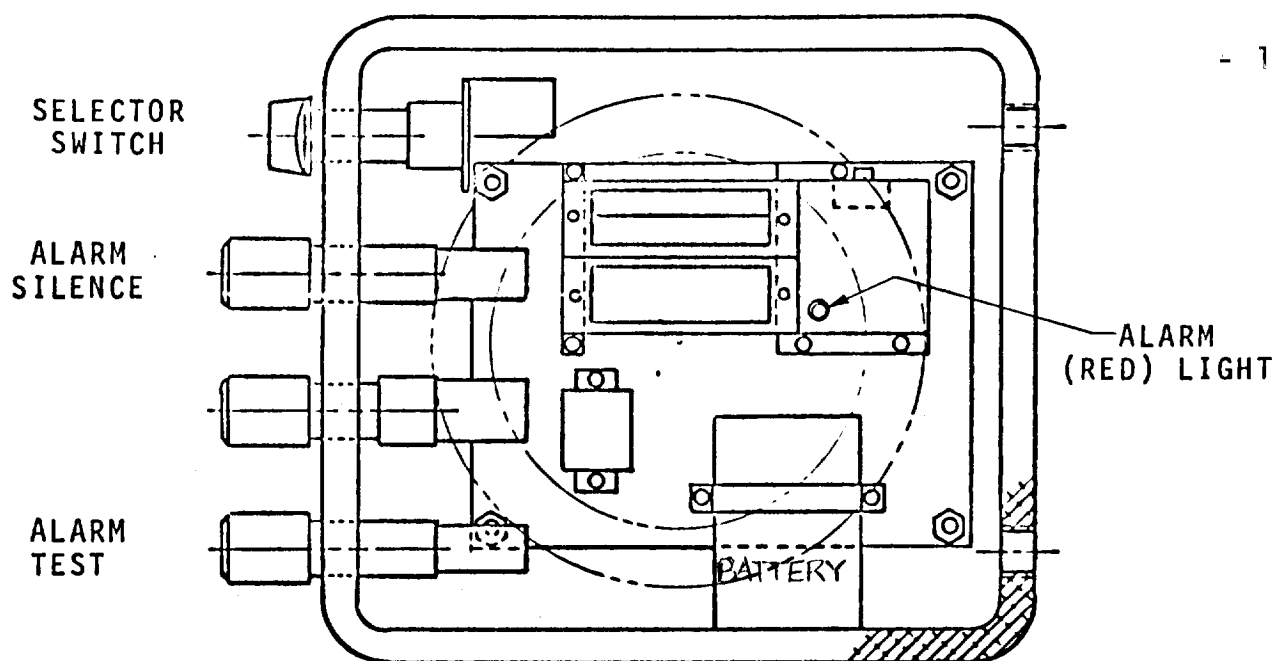


FIGURE 9
AUDIO-VISUAL ALARM LOCATION

5. Audio-Visual Alarm (TA-17) is activated by temperature above $-35^{\circ}\text{C}.$, as sensed by a thermocouple located on the exterior top of the inner tank, at the bottom of the dry ice chamber (Figure 1 and Figure 4). The alarm will only activate when the selector switch (top control on left side of junction box, as shown on Figure 9 above) is in the "on" position. The top of the three pushbuttons beneath the selector switch is used to "silence" the alarm; the pushbutton may be released, and the alarm will remain silent. The alarm light (Figure 9) remains on after the alarm has been silenced. The bottom of the three pushbuttons may be used to test the alarm, as the alarm will sound while the pushbutton is held in.

SPECIFICATIONS:

Audible Alarm -- Mallory Sonalert Signal #SC628P

Visual Alarm -- Drake Gemlite #5160-458-604, color red.

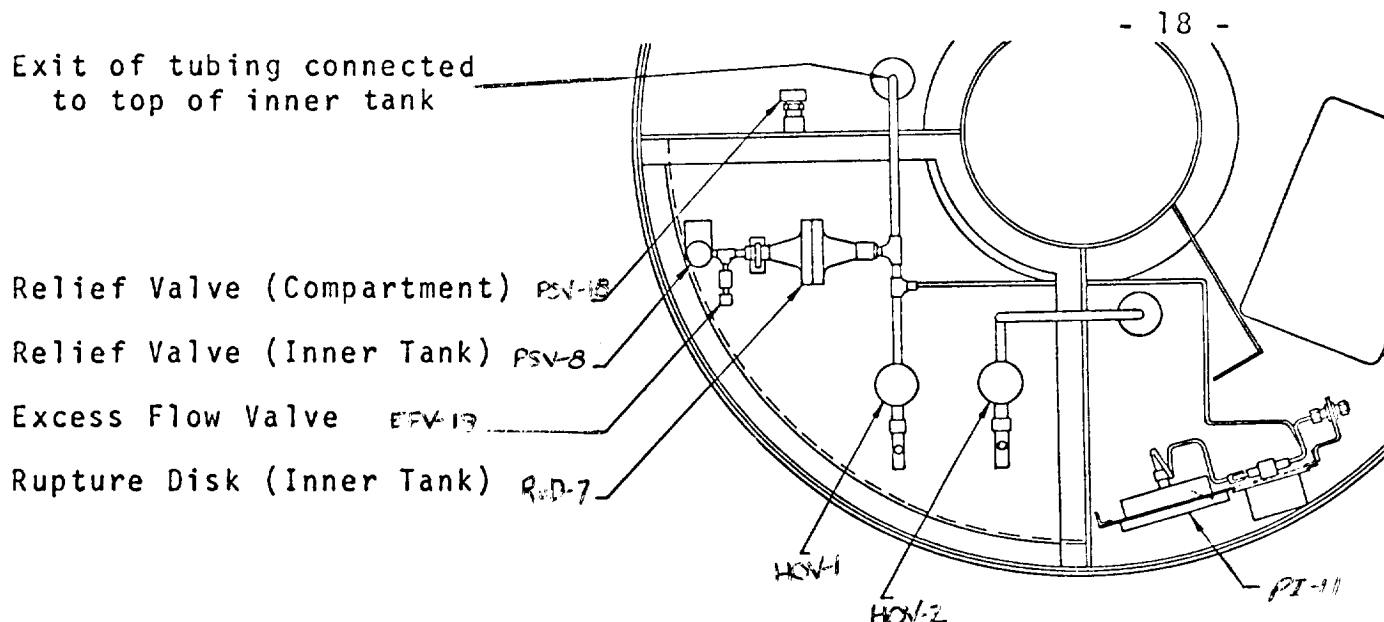


FIGURE 10
LOCATION OF SAFETY RELIEF DEVICES

6. Safety Relief Devices are shown in Figure 10 above; as well as in Figure 2 and Figure 4 (schematic diagram). The inner tank rupture disk (RuD-7) and the inner tank relief valve (PSV-8) are connected in series, with an excess flow valve (EFV-19) between to prevent pressure buildup downstream of the rupture disk. Compartment relief valve PSV-18 protects against excessive pressure buildup due to carbon dioxide vented to the valve compartment from the dry ice chamber.

SPECIFICATIONS:

- RuD-7 -- Rupture Disk Flange Assembly, 600#, with 18-8 studs, nuts, & lock washers. Assembly F, Fike Metal Products. Rupture Disk, prebulged, 1/2", 321 SST with Teflon coating on pressure side; burst pressure, 550 psig.
- PSV-8 -- Safety Valve with 1/2" FNPT inlet, Kunkle Figure 663-C, set at 550 psig.
- EFV-19-- Excess Flow Valve, 1/4" NPT with 303 SST body, 18-8 SST ball, and SST lock screw. Figure 27, Fike Metal Prod.
- PSV-18-- Relief Valve, 1/2", Circle Seal Popoff #D524T-4M-0.5; set pressure, 0.5 psig.

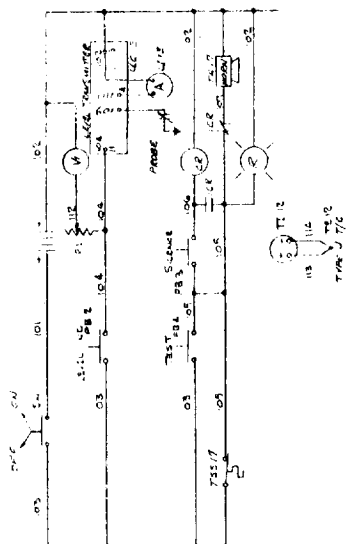
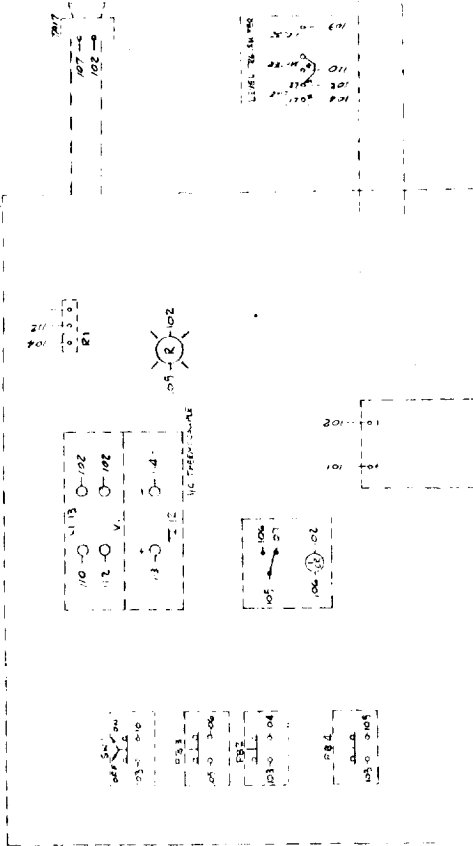
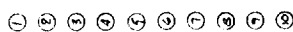


FIGURE 14
ELECTRICAL DETAILS
WIRING DIAGRAMS

AS BUILT

1971 2



THANK YOURS DIAGRAM

Current Date: 11/11/2018

- SCALE INFORMATION ON L13 IS CALCULATED FROM 1/3 THAT (3x) = 1/3. 1/3 HAS BEEN LOCATED IN THE ANALYSIS AND IDENTIFIED TO BE THE SAME AS THE CALCULATED SCALE IN THE ANALYSIS.
- SCALE FOR L15 UNDER 0-50 VOLTS
- SCALE FOR VI IS CALCULATED BY USING R1 AND VOLTMETER VI IS CALCULATED BY USING R1 AND A SEPARATE INSTRUMENT. SCALES ASSIGNED BY ENR
- TEST 17 TIME IN TEMS ALARM WITH CLOSER AT -35°C ON TEMP INCREASE
- ALL REQUIRED ATTENDING INSIDE CONTROL PANEL TO BE TENDERS WITH LOCKMEASURES IF LOCKTIME EXHAUST
- ALL MINS TO BE IDENTIFIED AT EACH TERMINATION WITH ROOM TIME INCREASES.
- ALL MINS TO BE IN ACCORDANCE WITH ALL C. REQUIRED ABOVE FOR CLASS 1, GROUP D

concupiscentia

PERFORMANCE

A. Storage/Shipment

Table 3 and Figure 15 give the results of a 30-day storage test with 200 pounds of diborane in the shipping container subsequently authorized under DOT Special Permit 6522. These data are presented for reference, as the normal thermal behavior to be expected.

Dry ice will be present for 13 to 14 days, after which time heat leak causes diborane temperature and pressure to rise slowly. At the 15-day maximum delivery time required by the permit, pressure is 60 psig. Recommended maximum operating pressure of 400 psig is reached during the 34th day.

B. Recooling

Figure 16 gives the results of recooling tests conducted immediately after the storage test described above. Again these data are presented for reference, as the normal thermal behavior to be expected.

After the third addition of dry ice, pressure dropped to 32 psig (which corresponds to a diborane temperature of $-70^{\circ}\text{C}.$) at 41 days, about 15 days (and 196 pounds of dry ice) after start of recooling.

TABLE 3

DIBORANE SHIPPING CONTAINER
30-DAY STORAGE TEST RESULTS

<u>TIME, DAYS</u>	<u>DRY ICE, POUNDS</u>	<u>PRESSURE PSIG</u>	<u>TEMP., °C</u>
0	108.0	18.0	-79.0
1	101.2	21.5	-76.0
2	93.8	24.2	-74.2
3	85.4	26.5	-72.8
4	77.0	27.7	-72.1
5	68.6	29.0	-71.4
6	60.2	30.3	-70.7
7	51.8	31.6	-70.0
8	43.4	32.9	-69.3
9	35.0	34.2	-68.6
10	26.6	35.5	-67.9
11	18.2	36.8	-67.2
12	10.6	38.1	-66.6
13	4.7	40.0	-65.9
14	0	45.7	-63.5
15	0	59.5	-58.3
16	0	74.5	-53.0
17	0	89.5	-48.6
18	0	104.5	-44.3
19	0	119.5	-40.5
20	0	135.5	-37.0
21	0	151.5	-33.7
22	0	167.6	-30.7
23	0	184.6	-27.7
24	0	202.2	-24.9
25	0	219.8	-22.1
26	0	238.6	-19.4
27	0	259.5	-16.6
28	0	280.5	-13.7
*29	0	301.5	-10.8
*30	0	322.4	- 7.9
*31	0	343.4	- 5.3
*32	0	364.4	- 2.8
*33	0	385.4	- 0.5
*34	0	406.3	+ 1.6

*Extrapolation

FIGURE 15

30-DAY STORAGE TEST RESULTS

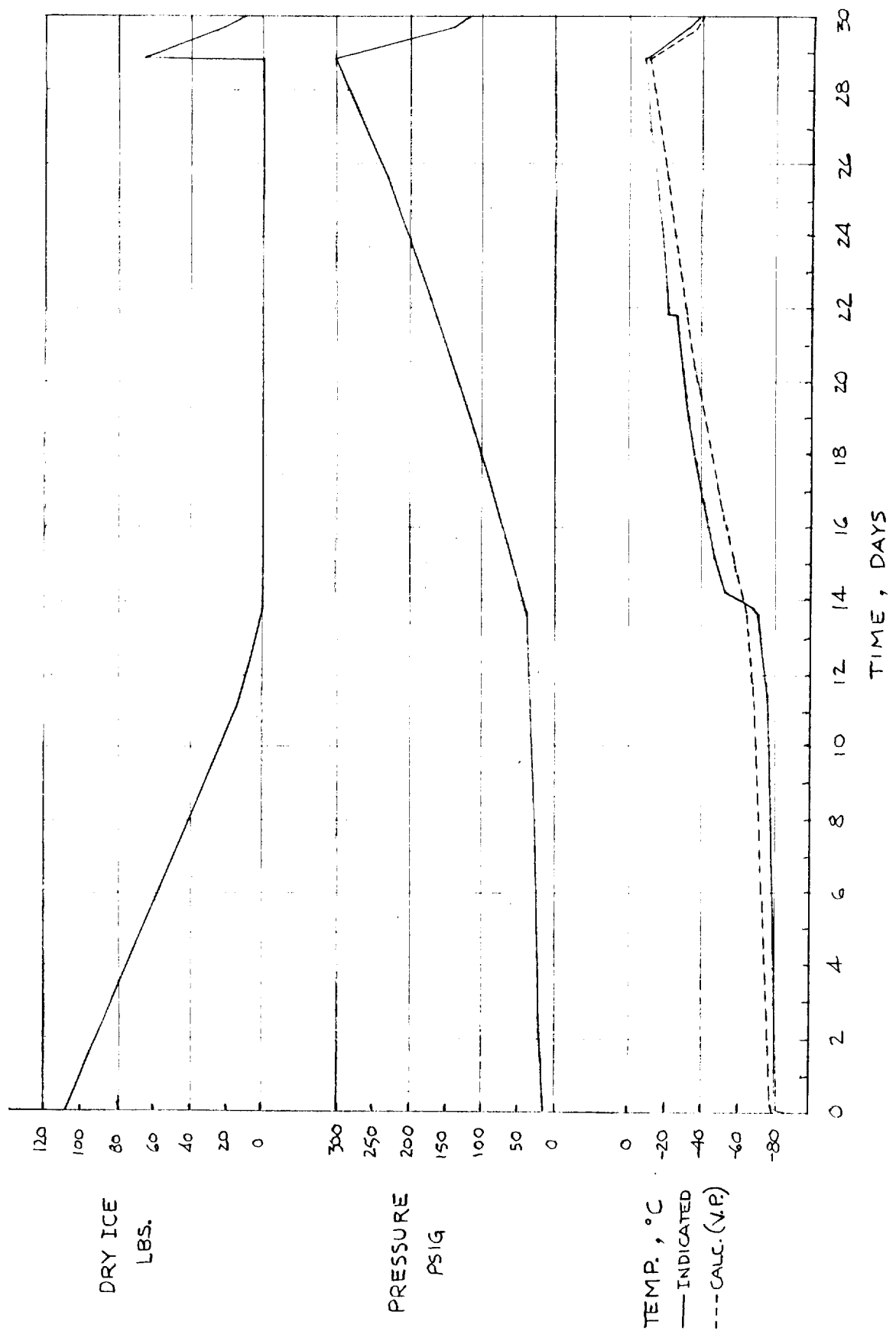
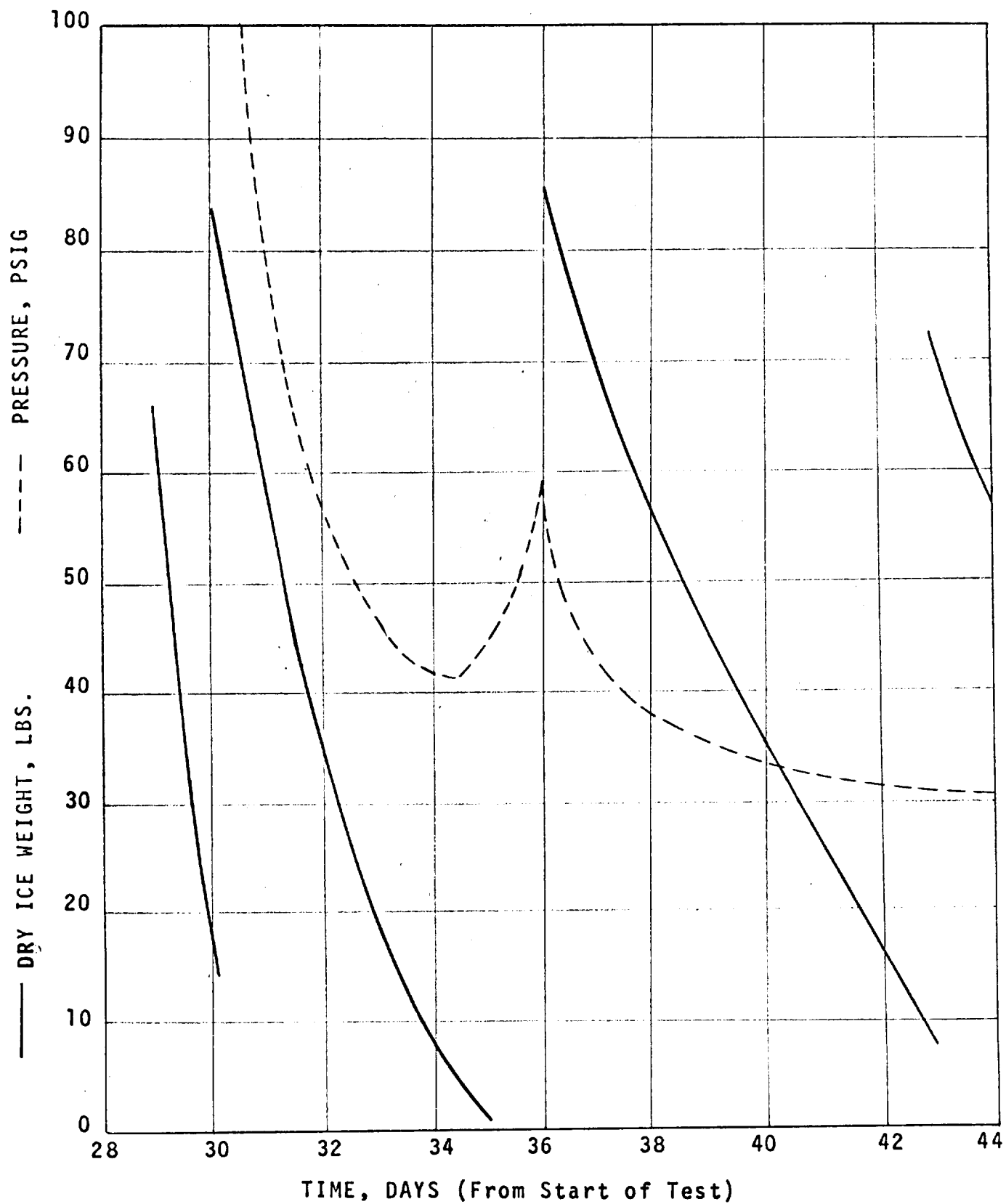


FIGURE 16
RECOOLING TEST RESULTS



SUMMARY OF PROVISIONS OF D.O.T. SP 6522
INCLUDING ALL REVISIONS THROUGH 20 OCTOBER 1972
AS COMPILED BY CALLERY CHEMICAL COMPANY

This special permit is issued pursuant to 49 CFR 170.15 of the Department of Transportation (DOT) Hazardous Materials Regulations, as amended, to authorize shipments of a poisonous and compressed gas; and a flammable liquid under conditions as prescribed herein. This permit does not relieve any shipper or carrier from compliance with any requirement of the DOT regulations, except as specifically provided for herein.

Standard special permit requirements and conditions relating to package markings, preparation of shipping papers, shipping experience reports, etc., are published in 49 CFR 171.6. These requirements are part of this special permit.

1. BASIS. September 22, 1972 petition by Jet Propulsion Laboratory, Pasadena, California.
2. COMMODITY. Diborane or methanol (methyl alcohol).
3. PROPER SHIPPING NAME (49 CFR 172.5).
 - a. For Diborane: Both "Compressed gas, n.o.s. (Flammable)" and "Poisonous gas, n.o.s.".
 - b. For Methanol: "Methanol" or "Methyl alcohol".
4. REGULATION WAIVED. 49 CFR SS173.119, 173.304 (a) (1) and 173.328.
5. AUTHORIZED SHIPPER. The petitioners identified above and their customers who register their identity with and receive acknowledgement from this Board and have a copy of the special permit.
6. PACKAGING PRESCRIBED. In a specially designed 36-inch diameter spherical inner tank having a maximum working pressure of 500 psig containing not less than 100 pounds nor more than 200 pounds of product. Inner tank is surrounded (except for the dry ice chamber) by a 48-inch diameter cylindrical shell containing perlite insulation. Container must be equipped with an audio-visual alarm to indicate temperature above minus 35°C. Container must be fabricated and assembled in accordance with details included in CVI Corporation's drawings A458-5800 through A458-5821 on file with this Board.

Except as otherwise provided herein, the container must be qualified and prepared for shipment in accordance with the Callery's design and performance specifications on file with this board.

- a. As an alternate to paragraph (6), shipments of diborane in quantities less than 100 pounds per container prescribed herein may be made subject to the following conditions:
 - i. The container and its contents must be precooled to minus 70°C. or lower, and the cooldown temperature must be confirmed by a measured ullage pressure of 32 psig or lower on the date of shipment.
 - ii. The container must have not less than 108 pounds of dry ice in the dry ice chamber on the day of shipment.
 - iii. The container must reach its destination within three days from the date of shipment.
 - iv. Shipments are limited to highway transportation via private carriage only.
 - v. All requirements of the permit, except as otherwise provided in this paragraph, are applicable.

7. SPECIAL PACKAGING REQUIREMENTS.

- a. All diborane shipments, including those which qualify as "empty" by subparagraph (c) must comply with the following:
 - i. In addition to the flammable gas label and the poison gas label, each outside shipping container must bear a conspicuous label reading as follows: "IF NOT DELIVERED BEFORE _____ CARRIER MUST ADVISE (Insert name and address of shipper), ALSO THE BUREAU OF EXPLOSIVES, WASHINGTON, D.C. BY WIRE." The date inserted in the blank space on this label must not be in excess of the number of days prescribed herein from the date shipment is offered for transportation.
 - ii. Container must have not less than 108 pounds of dry ice in dry ice chamber on the day shipment is made.
- b. All diborane shipments except those which qualify as "empty" by subparagraph (c) must also comply with the following:
 - i. Filling is to be by weight only.

- ii. Containers precooled to minus 80°C. with liquid nitrogen must reach destination within 15 days from date of shipment. True temperature at or below -80°C. must be confirmed by pressure of 18 psig or lower on date of shipment.
- iii. Containers cooled to minus 70°C. with dry ice (or liquid nitrogen) must reach destination within the following schedule.
 - (a) 15 days from date of shipment when diborane net weight is 175 to 200 pounds.
 - (b) 12 days from date of shipment when diborane net weight is 100 to 174 pounds.True temperature at or below -70°C. must be confirmed by pressure of 32 psig or lower on date of shipment.
- c. Container shipped as "empty" of diborane must also comply with the following:
 - i. Must be verified to be "empty" by one of the following:
 - (a) The empty weight must not exceed the marked tare weight by more than 5 pounds, or
 - (b) Level of liquid diborane must be below the bottom of the dip tube. Loss of liquid seal will be evident by ability to vent gas pressure from the container (to user's tank or other proper vent system) through the dip tube.
 - ii. Pressure should be vented to between 25 and 50 psig at the time container is emptied.
 - iii. Containers shipped as empty must reach destination within 15 days from date of shipment.
- d. Provisions for shipment of methanol are as follows:
 - i. The only time methanol may be shipped is to satisfy contractor's qualification acceptance tests. Under these conditions neither the transit time restrictions nor the requirement for precooling apply.
 - ii. Additionally, the requirements of subparagraphs (a), (b), and (c) of this paragraph and the entire paragraph (9) do not apply to methanol shipments.

8. MODES OF TRANSPORTATION AUTHORIZED. Motor vehicle.

9. SPECIAL TRANSPORTATION REQUIREMENTS.

- a. A copy of this permit, kept current, must be carried aboard each motor vehicle.
- b. Shipments of diborane made under the terms of this permit must be delivered within the time prescribed in subparagraph 7(b).
- c. Each shipping paper must show thereon, following the commodity description, the notation, appropriately executed:

DOT SPECIAL PERMIT NO. 6522
DATE OF SHIPMENT _____
IF NOT DELIVERED BEFORE _____
DAYS, CARRIER MUST ADVISE
BUREAU OF EXPLOSIVES, WASHINGTON,
D. C. BY WIRE.

- d. Each shipper must require acknowledgement of receipt of shipment from consignee by wire, to be confirmed in writing, and must promptly notify the Bureau of Explosives (AAR) of any such shipment not received at destination within two days after shipment is due.
- e. Any common carrier by motor vehicle transporting diborane under the terms of this permit must be specifically approved by the Federal Highway Administration.

10. REPORTING REQUIREMENTS. Any incident involving loss of contents of the package must be reported to this Board as soon as practicable.

11. EXPIRATION DATE. September 15, 1973.

Issued at Washington, D.C.:



DEPARTMENT OF TRANSPORTATION
HAZARDOUS MATERIALS REGULATIONS BOARD
WASHINGTON, D.C. 20590

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SPECIAL PERMIT NO. 6522
FIRST REVISION
(COMPLETE)

This special permit is issued pursuant to 49 CFR 170.15 of the Department of Transportation (DOT) Hazardous Materials Regulations, as amended, to authorize shipments of a poisonous and compressed gas; and a flammable liquid under conditions as prescribed herein. This permit does not relieve any shipper or carrier from compliance with any requirement of the DOT regulations, except as specifically provided for herein.

Standard special permit requirements and conditions relating to package markings, preparation of shipping papers, shipping experience reports, etc., are published in 49 CFR 171.6. These requirements are part of this special permit.

1. BASIS. March 15 and 28, 1972 petition by Callery Chemical Company, Callery, Pa.; March 20, 1972 petition by Jet Propulsion Laboratory, Pasadena, Calif.

2. COMMODITY. Diborane or methanol (methyl alcohol).

3. PROPER SHIPPING NAME (49 CFR 172.5).

a. For Diborane: Both "Compressed gas, n.o.s. (Flammable)" and "Poisonous gas, n.o.s.".

b. For Methanol: "Methanol" or "Methyl alcohol".

4. REGULATION WAIVED. 49 CFR §§173.119, 173.304(a)(1) and 173.328.

5. AUTHORIZED SHIPPER. The petitioners identified above and their customers who register their identity with and receive acknowledgement from this Board and have a copy of the special permit.

6. PACKAGING PRESCRIBED. In a specially designed 36-inch diameter spherical inner tank having a maximum working pressure of 500 psig containing not less than 100 pounds nor more than 200 pounds of product. Inner tank is surrounded (except for the dry ice chamber) by a 48-inch diameter cylindrical shell containing perlite insulation. Container must be equipped with an audio-visual alarm to indicate temperature above minus 35°C. Container must be fabricated and assembled

in accordance with details included in CVI Corporation's drawings A458-5800 through A458-5821 on file with this Board. Except as otherwise provided herein, the container must be qualified and prepared for shipment in accordance with the Callery's design and performance specifications on file with this Board.

7. SPECIAL PACKAGING REQUIREMENTS.

a. All diborane shipments, including those which qualify as "empty" by subparagraph (c), must comply with the following:

i. In addition to the flammable gas label and the poison gas label, each outside shipping container must bear a conspicuous label reading as follows: "IF NOT DELIVERED BEFORE _____ CARRIER MUST ADVISE (Insert name and address of shipper), ALSO THE BUREAU OF EXPLOSIVES, WASHINGTON, D.C., BY WIRE." The date inserted in the blank space on this label must not be in excess of the number of days prescribed herein from the date shipment is offered for transportation.

ii. Container must have not less than 108 pounds of dry ice in dry ice chamber on the day shipment is made.

iii. Container must reach destination within 15 days from date of shipment.

b. All diborane shipments except those which qualify as "empty" by subparagraph (c) must also comply with the following:

i. Filling is to be by weight only.

ii. Container must be precooled with liquid nitrogen to below minus 80°C. and excess nitrogen removed prior to charging with diborane.

c. Container shipped as "empty" of diborane must also comply with the following:

i. Must be verified to be "empty" by one of the following:

(a) The empty weight must not exceed the marked tare weight by more than 5 pounds, or

- (b) Level of liquid diborane must be below the bottom of the dip tube. Loss of liquid seal will be evident by ability to vent gas pressure from the container (to user's tank or other proper vent system) through the dip tube. Pressure should be vented to between 25 and 50 psig at the time container is emptied.

d. Provisions for shipment of methanol are as follows:

- i. The only time methanol may be shipped is to satisfy contractor's qualification acceptance tests. Under these conditions neither the transit time restrictions nor the requirement for precooling apply.
- ii. Additionally, the requirements of subparagraphs (a), (b), and (c) of this paragraph and the entire paragraph (9) do not apply to methanol shipments.

8. MODES OF TRANSPORTATION AUTHORIZED. Motor vehicle.

9. SPECIAL TRANSPORTATION REQUIREMENTS.

- a. A copy of this permit, kept current, must be carried aboard each motor vehicle.
- b. Shipments of diborane made under the terms of this permit must be delivered within 15 days from date of shipment.
- c. Each shipping paper must show thereon, following the commodity description, the notation, appropriately executed:

DOT SPECIAL PERMIT NO. 6522
DATE OF SHIPMENT _____
IF NOT DELIVERED BEFORE _____
DAYS, CARRIER MUST ADVISE
BUREAU OF EXPLOSIVES, WASHINGTON,
D.C. BY WIRE.

- d. Each shipper must require acknowledgement of receipt of shipment from consignee by wire, to be confirmed in writing, and must promptly notify the Bureau of Explosives (AAR) of any such shipment not received at destination within two days after shipment is due.

Continuation of 1st Rev SP 6522

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e. Any common carrier by motor vehicle transporting diborane under the terms of this permit must be specifically approved by the Federal Highway Administration.

10. REPORTING REQUIREMENTS. Any incident involving loss of contents of the package must be reported to this Board as soon as practicable.

11. EXPIRATION DATE. September 15, 1972.

Issued at Washington, D.C.:

for B. R. Fiste
W. R. Fiste
For the Administrator
Federal Highway Administration

17 APR 1972
(DATE)

Address all inquiries to: Secretary, Hazardous Materials Regulations Board, U.S. Department of Transportation, Washington, D.C. 20590. Attention: Special Permits.

Dist: a, d



DEPARTMENT OF TRANSPORTATION
HAZARDOUS MATERIALS REGULATIONS BOARD
WASHINGTON, D.C. 20390

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SPECIAL PERMIT NO. 6522
SECOND REVISION

Pursuant to 49 CFR 170.15 of the Department of Transportation (DOT) Hazardous Materials Regulations, as amended, and on the basis of the April 21 and August 30, 1972 petitions by Callery Chemical Co., Callery, Pennsylvania.

Special Permit No. 6522 is hereby amended by deleting subparagraph (7(a)(iii)); amending subparagraphs (7(b)(ii)), (7(c)(i)(b)), (9b) and paragraph (11); adding subparagraphs (7(b)(iii)) and (7(c)(ii) and (iii)) as follows:

"7a. * * *

i. * * *

ii. * * *

iii. Deleted.

"7b. * * *

i. * * *

ii. Containers precooled to minus 80°C. with liquid nitrogen must reach destination within 15 days from date of shipment. True temperature at or below -80°C. must be confirmed by pressure of 18 psig or lower on date of shipment.

iii. Containers cooled to minus 70°C. with dry ice (or liquid nitrogen) must reach destination within the following schedule.

(a) 15 days from date of shipment when diborane net weight is 175 to 200 pounds.

(b) 12 days from date of shipment when diborane net weight is 100 to 174 pounds.

True temperature at or below -70°C. must be confirmed by pressure of 32 psig or lower on date of shipment.

"7c. * * *

i. * * *

(a) * * *

Continuation of 2nd Rev. SP 6522

Page 2

(b) Level of liquid diborane must be below the bottom of the dip tube. Loss of liquid seal will be evident by ability to vent gas pressure from the container (to user's tank or other proper vent system) through the dip tube.

ii. Pressure should be vented to between 25 and 50 psig at the time container is emptied.

iii. Containers shipped as empty must reach destination within 15 days from date of shipment.

"9a. * * *

"9b. Shipment of diborane made under the terms of this permit must be delivered within the time prescribed in subparagraph 7(b).

"11. EXPIRATION DATE. September 15, 1973."

All other terms of this permit, as revised, remain unchanged. The complete permit currently in effect consists of the First and Second Revisions.

Issued at Washington, D.C.:



W. R. Fiste
For the Administrator
Federal Highway Administration

2-14-72
(DATE)

Address all inquiries to: Secretary, Hazardous Materials Regulations Board,
U. S. Department of Transportation, Washington, D.C. 20590.
Attention: Special Permits.

Dist: a, d



DEPARTMENT OF TRANSPORTATION
HAZARDOUS MATERIALS REGULATIONS BOARD
WASHINGTON, D.C. 20590

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SPECIAL PERMIT NO. 6522
THIRD REVISION

Pursuant to 49 CFR 170.15 of the Department of Transportation (DOT) Hazardous Materials Regulations, as amended, and on the basis of the September 22, 1972 petition by Jet Propulsion Laboratory, Pasadena, California:

Special Permit No. 6522 is hereby amended by adding paragraph (6a) to read as follows:

"6a. As an alternate to paragraph (6), shipments of diborane in quantities less than 100 pounds per container prescribed herein may be made subject to the following conditions:

i. The container and its contents must be pre-cooled to minus 70°C. or lower, and the cooldown temperature must be confirmed by a measured ullage pressure of 32 psig or lower on the date of shipment.

ii. The container must have not less than 108 pounds of dry ice in the dry ice chamber on the day of shipment.

iii. The container must reach its destination within three days from the date of shipment.

iv. Shipments are limited to highway transportation via private carriage only.

v. All requirements of the permit, except as otherwise provided in this paragraph, are applicable.

All other terms of this permit, as revised, remain unchanged. The complete permit currently in effect consists of the First, Second and Third Revisions.

Issued at Washington, D.C.:

for B.A. Milster
W. R. Fiste
For the Administrator
Federal Highway Administration

20 OCT 1972
(DATE)

Continuation of 3rd Rev. SP 6522

Page 2

Address all inquiries to: Secretary, Hazardous Materials
Regulations Board, U.S. Department of Transportation,
Washington, D.C. 20590. Attention: Special Permits.

Dist: a, d
Jet Propulsion Laboratory, Pasadena, Ca.
North American Rockwell, Canoga Park, Ca.
Callery Chemical Co., Callery, Pa.

CCC 72-3

A. DIBORANE PROPERTIES

TABLE 4
DIBORANE PHYSICAL PROPERTIES

Formula	B ₂ H ₆
Molecular Weight	27.668
Purity	Typically at least 99 mole per cent
Color and State	Colorless gas at room temperature and one atmosphere
Odor	Unpleasant, distinctive
Melting Point	-164.85°C. (-264.73°F.)
Boiling Point	- 92.6°C. (-134.7°F.)
Density, (See Figure 17)	
solid (at -183°C.)	0.577 g/ml (36.02 lbs/ft ³)
liquid (at -92.6°C.)	0.436 g/ml (27.21 lbs/ft ³)
Critical Temperature	+16.7°C. (+62.1°F.)
Critical Pressure	39.5 atmospheres (581 psia)
Surface Tension,	
liquid (at -92.6°C.)	13.45 dynes/cm (9.22 X 10 ⁻⁴ lb-f/ft)
	$\gamma = 72.07 - 0.5152T + 1.055 \times 10^{-3}T$ where γ = surface tension, dynes/cm T = temperature, °K
	$\gamma = 4.939 \times 10^{-3} - 1.961 \times 10^{-5}T + 2.232 \times 10^{-8}T^2$ where γ = surface tension, lb-f/ft T = temperature, °R
Vapor Pressure (See Figure 18)	
	$\log P = 5.1104 - \frac{1157.74}{T - 31.4}$ where P = pressure, psia T = temperature, °R
Viscosity,	
liquid (at -92.6°C.)	0.131 centipoise
gas (at +30°C.)	0.008 centipoise

CCC 72-3

Dielectric Constant liquid	1.8727 @ -92.6°C.
gas	1.0018 @ STP
Heat of Fusion, (at -164.85°C.)	1069 cal/mole (69.55 BTU/lb.)
Heat of Vaporization (at -92.6°C.)	3413 cal/mole (222.0 BTU/lb.)
Heat of Formation gas (at 25°C.)	9.8 kcal/mol (637.5 BTU/lb.)
liquid (at -92.6°C.)	5.0 kcal/mol (325 BTU/lb.)
Heat of Combustion, net	-478.4 kcal/mole (31,100 BTU/lb.)

NOTE: For a compilation of complete diborane properties, see
DIBORANE HANDBOOK (N70-78384) by M. T. Constantine et al.
of the Research Division of Rocketdyne (a Division of
North American Rockwell Corporation) prepared for NASA
under Contract NAS7-769.

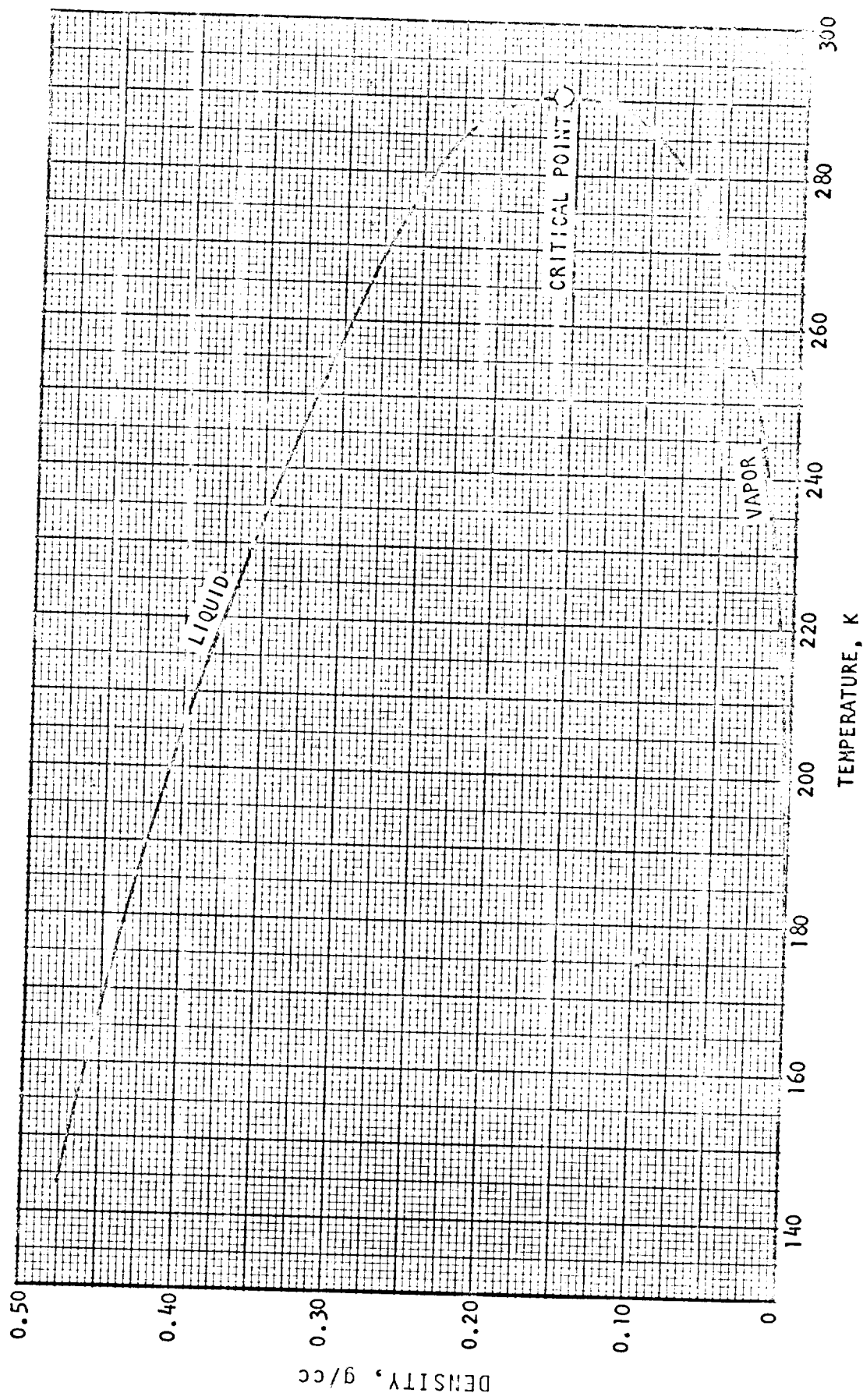


FIGURE 17

DENSITY OF SATURATED LIQUID AND VAPOR DIBORANE

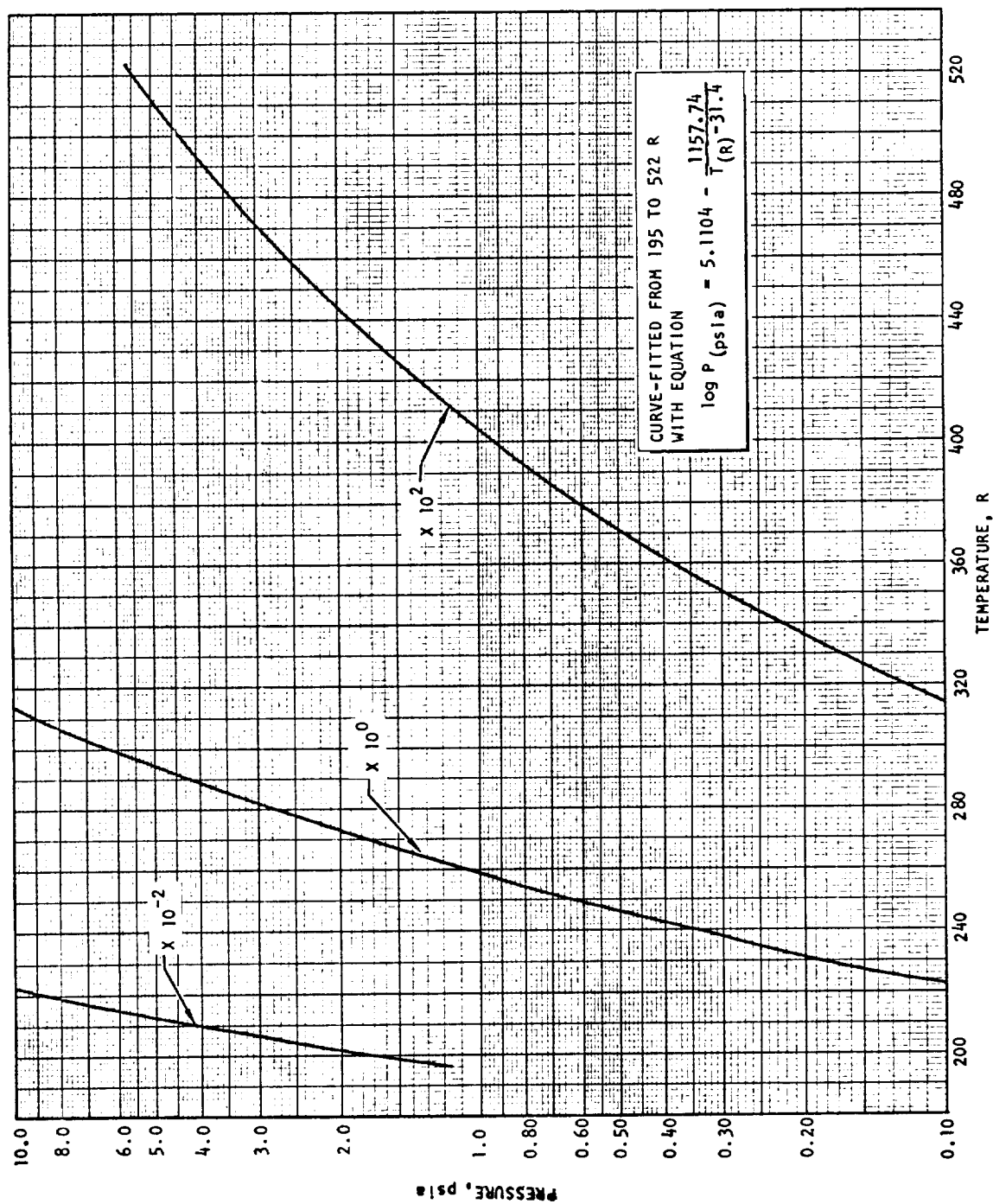


FIGURE 18

VAPOR PRESSURE OF DIBORANE

B. SAFETY AND HANDLING

NOTE: The HAZARDS Section below is reprinted from the DIBORANE HANDBOOK compiled for NASA. The remainder of this section is abstracted from the same reference.

6.1 HAZARDS

The potential hazards involved in the use of B_2H_6 are associated with three of the general health hazard categories: toxicity (physiological activity), flammability, and explosivity. Diborane is a highly toxic chemical and serious personnel health hazards can result from inhalation or ingestion of, or cutaneous exposure to, its liquid and/or vapors and many of its reaction and decomposition products. It is classified as a "Flammable Compressed Gas" * (Red Label) by the Department of Transportation and will almost always ignite on contact with air. Although B_2H_6 will undergo rapid decomposition when subjected to extreme temperatures and can form explosive mixtures with air (like most fuels), it is not sensitive to mechanical shock and does not present an unusual explosion hazard. While these hazards may sound ominous, it should be noted that similar hazards exist for many chemical compounds which are in widespread use and are safely handled by industry. Thus, adequately trained personnel with full knowledge of the potential hazards and their control and following prescribed procedures can safely handle B_2H_6 in any required operation.

*In December, 1971 the Department of Transportation Hazardous Materials Regulation Board, through issue of Special Permit No. 6522, classified diborane as

"Compressed gas, n.o.s. (Flammable)"
and
"Poisonous gas, n.o.s."

HAZARD CONTROL

The probability of a B_2H_6 leak is always present. A B_2H_6 leak creates fire, explosion, and toxicity hazards. Continual monitoring for instantaneous detection and immediate action are necessary for effective control.

Hazard Detection

A large B_2H_6 leak will probably result in fire or explosion; a small leak may just oxidize slowly. Detection of leaks is necessary for protection from fire, explosion, and toxic hazards.

Odor must not be relied upon for detection. B_2H_6 storage and handling areas should be monitored with selective vapor detectors and an alarm system. Very small leaks should be suspected if a white powder is formed at pipe joints or around valve stems.

Devices are available to determine boron hydride concentrations in air (MSA Portable Borane and MSA Billion-Aire Detectors). No B_2H_6 selective instrument is currently available.

Leakage and Spill Control

On detection of a B_2H_6 leak:

1. Stop any B_2H_6 transfer or handling operation.
2. Close valves (remotely if possible) to isolate B_2H_6 tank.
3. Locate source of the leak.
4. Close valves to isolate affected components.
5. Dispose of B_2H_6 in affected components.
6. Replace or repair the leaking component.

B_2H_6 evaporates very rapidly. Primary concern is toxic hazard and fire. Action depends on quantity, weather, and location. Control by burning the B_2H_6 or hosing with water. Monitor toxic vapors.

Fire and Explosion Control

Leaks or spills of B_2H_6 will usually result in fires (with possibility of explosion). The best way to fight a B_2H_6 fire is (1) shutoff diborane source and (2) let it burn out while minimizing damage.

The fire should be kept under control by water fog, but should not be extinguished until the source of B_2H_6 is

valved off, isolated, or depleted. Contain with deluge systems and water spray to prevent its spread.

The most effective extinguishing agent is water fog, which contains the fire and hydrolyzes the B_2H_6 .

Carbon dioxide may be used for small fires. Dry chemicals and foams are ineffective.

WARNING

DO NOT USE CARBON TETRACHLORIDE

Halogenated hydrocarbons form impact sensitive mixtures with B_2H_6

Personnel fighting B_2H_6 fires must use respiratory equipment; in an enclosed area, self-contained breathing equipment is mandatory.

Decontamination

The area can be decontaminated by washing with a water solution containing a mixture of 5-percent ammonia and 5-percent O-B detergent (e.g., trisodium phosphate). After 30 to 60 minutes the materials should be washed into an approved drainage system with copious amounts of water. Any boric oxide and/or boric acid can also be flushed readily with water.

Equipment should also be decontaminated prior to handling. Immerse the contaminated portions in water or water-methanol solution for a short time, then flush with water and dry.

Any personnel exposed to the smoke or residual solutions should be decontaminated immediately. Clothing should be removed and laundered prior to reuse. Body areas in contact with contaminated clothing should be scrubbed with 1- to 5-percent aqueous triethanolamine or with 3-percent aqueous ammonium hydroxide, followed by soap and water. Avoid eye contact with wash solutions. Respiratory protection should be continued until the contaminated clothing is removed and the body has been washed.

FACILITY AND PERSONNEL PROTECTION

Safety Equipment

Toxicity and flammability of B_2H_6 dictate the need for adequate safety equipment to protect operating personnel and facilities.

Facility Safety Equipment

Equipment for facility protection should consist of safety showers, eye baths, a water deluge system, fire blankets, portable fire extinguishers, fire hoses, vapor

detectors, and first-aid kits. This equipment should be strategically located and easily accessible.

All operating personnel should be thoroughly familiar with the location and operation of each piece of safety equipment. The operating condition of the equipment must be verified periodically.

Personnel Protective Equipment

All personnel in the B_2H_6 handling and storage areas must wear flame-retardant clothing at all times. All personnel performing B_2H_6 transfer operations should wear fully protective personnel equipment.

Respiratory Protection*

Respiratory protective devices must be a part of the safety clothing for all personnel who might be exposed to boron hydride vapors. Only air-line respirators, hose masks and self-contained breathing apparatus are recommended. Air purifying devices such as gas masks and chemical cartridge respirators should not be used because of limited protective capability and because the odor threshold of diborane exceeds the threshold limit value.

*This section contains the recommendations of Callery Chemical Company, not an abstract of the NASA DIBORANE HANDBOOK.

Air supplied and self-contained devices should be approved by the U. S. Bureau of Mines and/or the National Institute of Occupational Safety and Health. The American National Standards Institute Practices for Respiratory Protection (ANSI Z 88.2) should be referred to for guidance in establishing an effective respiratory protection program.

Head and Face Protection

Hard hats or head coverings which cannot be penetrated by boron hydrides are recommended.

Safety glasses with side shields should be used where continuous eye protection is required. Full-length plastic face shields should also be worn when complete face protection is necessary.

Foot Protection

Safety shoes with built-in steel toe caps are recommended. Rubber boots should be used in contaminated areas.

Skin Protection

Personnel who may contact B_2H_6 must wear protective clothing. Flame-proofed coveralls made of Gra-lite and neoprene-dipped or Gra-lite gloves are suggested. All contaminated clothing must be changed immediately and laundered before reuse.

First Aid and Self Aid

IF B_2H_6 CONTACTS THE SKIN: The affected areas should be washed immediately with 1- to 5- percent aqueous tri-ethanolamine or with 3- percent aqueous ammonium hydroxide, followed by soap and water. All contaminated clothing should be promptly removed. If burns are present, refer to a physician.

IF B_2H_6 CONTACTS THE EYES: The eyes should be flushed immediately with water for at least 15 minutes and medical assistance should be obtained.

IF B_2H_6 (SOLUTIONS) ARE INGESTED: Every effort should be made to induce vomiting by administering large quantities of salt water or baking soda solution. A physician must be called immediately.

IF B_2H_6 VAPORS ARE INHALED: The exposed individual should be removed immediately from further exposure, kept quiet, and be placed in the care of an authorized physician

as soon as possible. If breathing stops, artificial respiration (or oxygen) should be administered. If indications of intoxication are observed, oxygen should be administered by mask for periods up to 1 hour. Bed rest is essential to rapid recovery of more severe cases and should be enforced judiciously. Never attempt to give anything by mouth to an unconscious patient.

Medical Treatment

IN ALL CASES OF EXTREME EXPOSURE OR CONTACT, A PHYSICIAN SHOULD BE NOTIFIED. The physician should be previously informed and educated as to the symptoms and treatment of B₂H₆ intoxication.

V PROCEDURE FOR CLEANOUT

WARNING: DIBORANE SAFETY AND HANDLING INFORMATION IN SECTION III OF THIS MANUAL MUST BE READ AND UNDERSTOOD BEFORE PROCEEDING WITH ANY OPERATIONS INVOLVING DIBORANE.

A. Receiving "Empty" Container

1. Unloading Container from Truck

The container may be moved either by fork lift truck or by crane. The following precautions should be noted:

a. Fork Lift Truck

- (1) Use a fork lift truck with capacity for at least 3000 pounds at a distance of 30 inches. Fork length must be at least 32 inches, and spread of forks should be at least 30 inches outside-to-outside.
- (2) Follow all standard safety practices for use of fork lift trucks. Be sure to block both front and rear wheels of truck from which container is removed before driving fork lift onto bed of truck.

- (3) The weight coming off the truck will be 2900 pounds plus the weight of the fork lift truck. Suitable supports or tiedowns may be necessary to prevent tipping up of the truck.

b. Crane

- (1) Use a crane with capacity for at least 3000 pounds at the distance the container will be handled. Use a spreader bar to separate drop chains at least 50 inches; drop chains should be at least 30 inches long.
- (2) Follow all standard safety practices for use of cranes.

2. Action Required on Date Received:

- a. Immediately after receiving the container, pressure should be checked to verify a safe condition. Pressure may be checked by reading the gauge (PI-11) through the plexiglass window, without removing the side panel. The gauge shutoff valve (HOV-15) is left open* during shipment.
- b. If necessary, setup and vent excessive pressure from the container.

*At the earliest opportunity, remove side panel and confirm that the valve is open (so that the gauge does in fact read the actual container pressure).

(1) Pressure should not reach 400 psig unless an error was made by the shipper (failure to vent inert gas after pressurization, or return of diborane net weight in excess of 5 pounds). In the event pressure should be over 400 psig, arrangements should be made for safe venting immediately. Pressure should be monitored continually, keeping in mind that pressure relief devices operate at 550 psig.

(2) It is recommended that the container be vented the same day if pressure is over 200 psig.

c. Inspect container for evidence of any damage in shipment. Carefully inspect piping for white solids deposits as evidence of slight leakage.

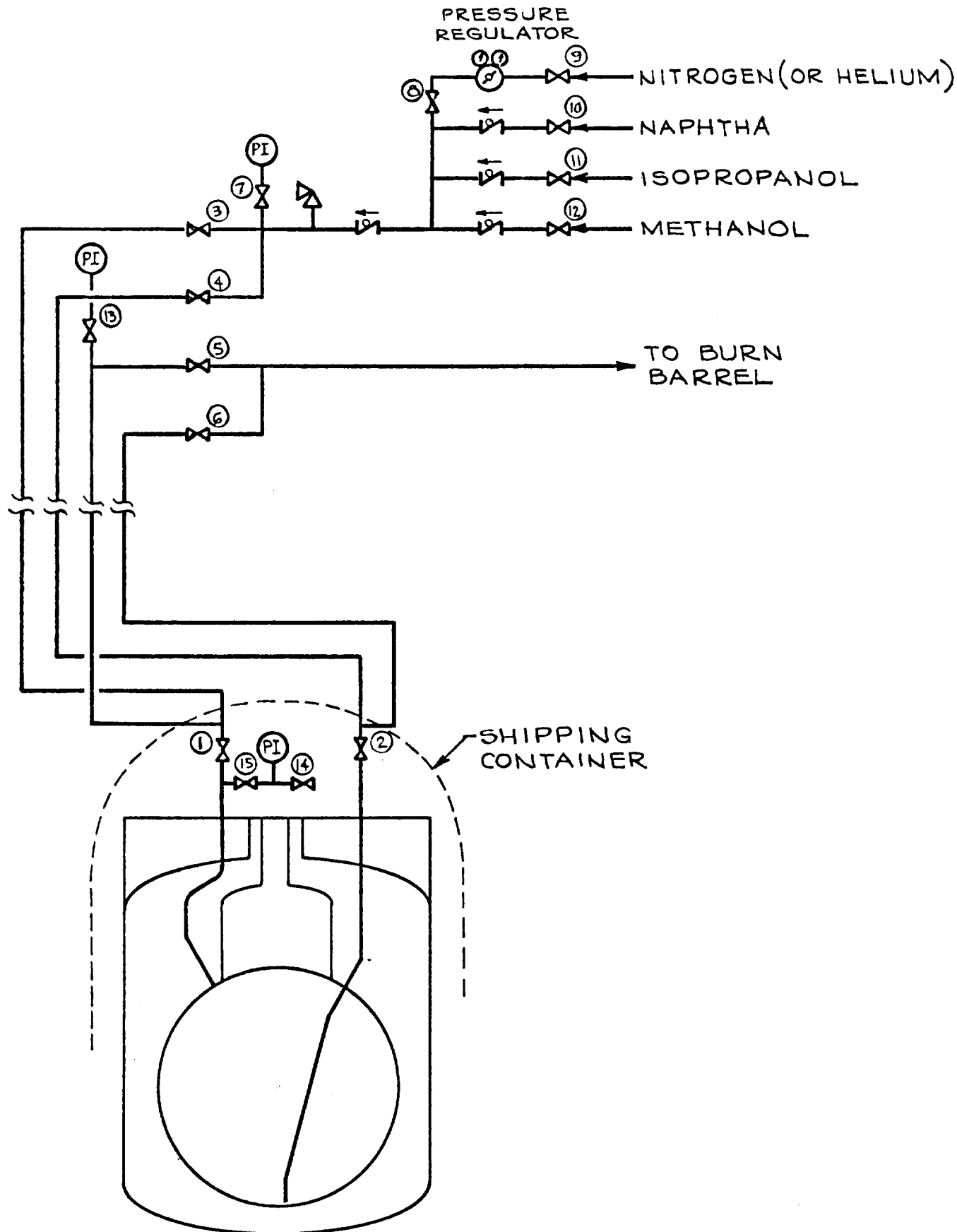
d. Schedule washout of container as soon as convenient.

B. Container Washout

1. Refer to Figure 19 for a Schematic Diagram of Cleanout Facility, showing valve numbers used in this procedure.

FIGURE 19

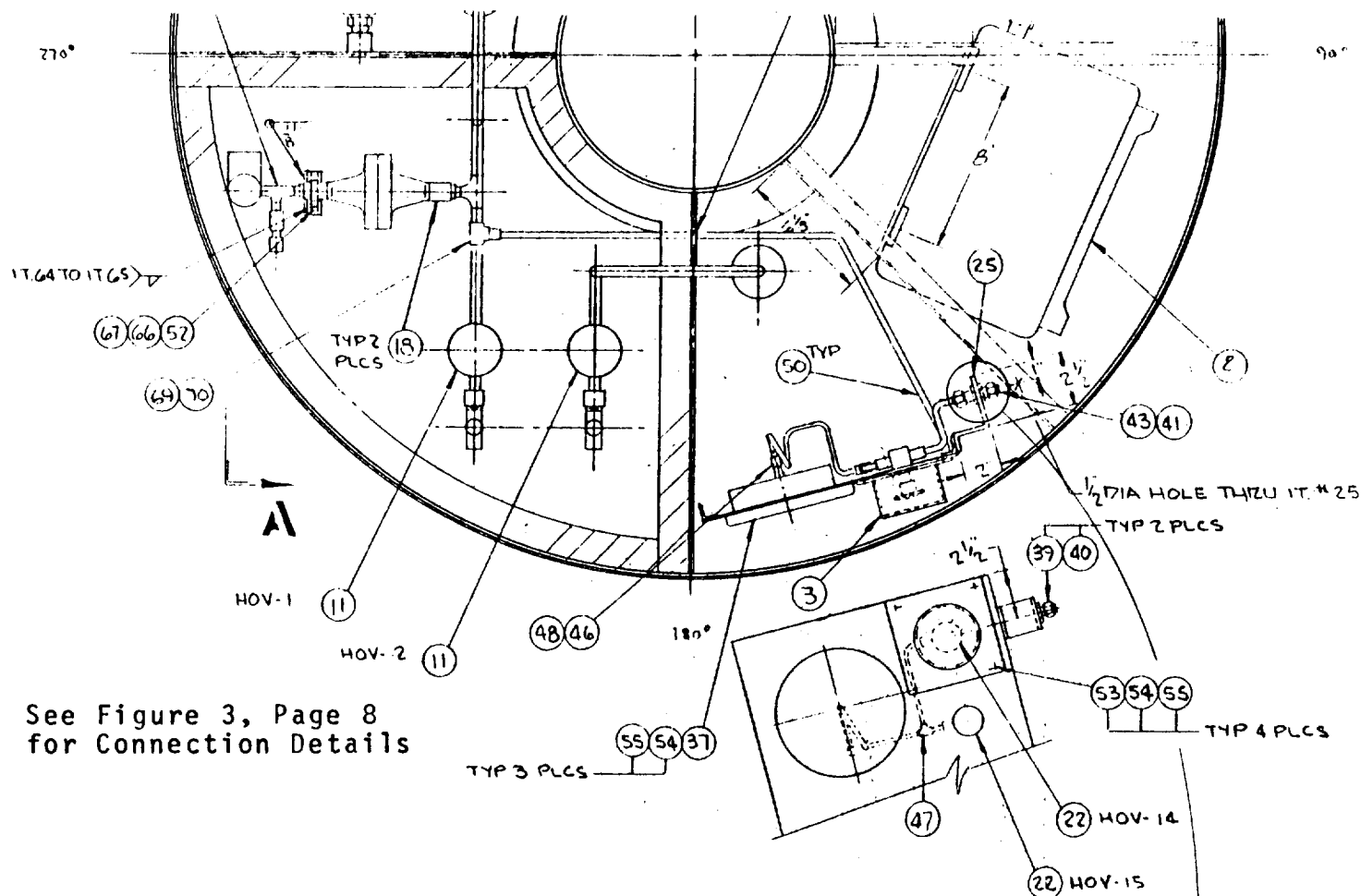
SCHEMATIC DIAGRAM OF CLEANOUT FACILITY



2. Remove thermal plug to confirm that dry ice is all gone. Replace plug and check weight of container. Refer to tare weight to determine weight of diborane remaining.
3. Connect the diborane shipping container to a piping system equivalent to that shown in Figure 19. This facility should be located in an appropriately remote area.*
 - a. Connect 1/4-inch tubing (copper or stainless steel) to gauge purge connection. Refer to Figure 20 on the next page for location and connection details.
 - b. Remove valve cover from gauge purge valve, HOV-14. Refer to Figure 20 for details.
 - c. Connect container vent valve (HOV-1) and container dip tube valve (HOV-2) to cleanout facility piping. Location of valves is shown in Figure 20. Connection details were given in Figure 3 on Page 8.
4. Setup to vent residual diborane from container, either into a barrel of burning solvent (such as naphtha or kerosene) or to another suitable disposal system.*

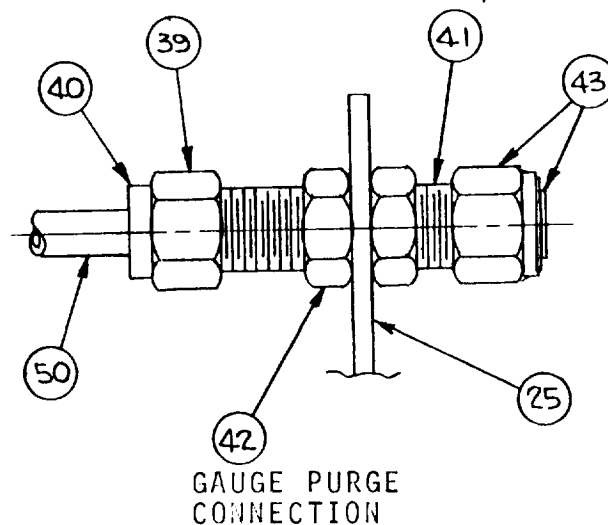
*See sections 4.5.5 and 4.5.6 of the DIBORANE HANDBOOK prepared for NASA by Rocketdyne for other applicable comments.

FIGURE 20
LOCATION OF CONTAINER VALVES AND
CONNECTIONS USED FOR CLEANOUT



See Figure 3, Page 8
for Connection Details

- 2 Junction Box
- 3 Valve Cover
- 22 1/4" Hand Oper. Valve w/Tube Soc. Weld Ends, 1/4" OD Tube; Nupro # 55-4H-SW
- 25 304 S.S. Sheet, 0.109" X 1-1/2" X 6"
- 37 Pressure Gauge, 4-1/2" Dial, 30" Vac to 0 psig to 1000 psig Range, Flush Mtg., 1/2" Back Conn., Ashcroft Maxisafe #1377SS
- 39 7/16" - 20 UNF-3B Union Nut AN 805-C4, 304 S.S.
- 40 1/4" OD Sleeve Coupling AN 819-4C, 304 S.S.
- 41 7/16" 20 NF-3 Union, 1/4" Tubing AN 832-4, 304 S.S.
- 42 7/16" 20 NF-3 Nut, 1/4" Tubing AN 924-4C, 304 S.S.
- 43 1/4" OD Cap Assembly, AN 929-4C, 304 S.S.
- 46 1/2" - 1/4" NPT Hex Reducing Cplg., Cajon #B-HRCG-4-316
- 47 1/4" OD Socket Weld Tee, Cajon #4SW-3, 304L
- 48 1/4" OD Male Conn. Swagelok #400-1-4-316
- 50 1/4" OD X .035" Wall Tubing, 304 ELC S.S.
- 53 #10-24 UNC Rd Hd Mach Scr. X 1/2" lg., Cad Plt. C.S.
- 54 #10-24 UNC Hex Nut, Cad Plt. C.S.
- 55 #10 Spring Lockwasher, Cad Plt. C.S.
- 69 1/2" OD Socket Weld Tee, Cajon #8SW-3-304L
- 70 1/4" OD Socket Weld Union, Cajon #4 SW-6-304L



5. This procedure assumes that all valves on Figure 19 are closed at the start, except gauge shutoff valves (7), (13), and (15).
6. Open nitrogen supply through valves (9) and (8). Open nitrogen through valves (3) and (4) up to container connections, but do not open valves (1) and (2) yet.
7. Test connections with soap solution to be sure there are no leaking joints.
8. Open valve (6) and purge nitrogen through for 3 to 5 minutes*; then close valve (6) and valve (4).
9. Open valve (5) and purge nitrogen through for 3 to 5 minutes*; then close valve (5) and valve (3).
10. Recheck that valves (3) and (5) are closed; then open container valve (1). Check that line pressure gauge reads the same as container pressure gauge.
11. Begin venting pressure from the container by gradually opening valve (5). Use valve (5) to control venting rate within the capacity of the system for safe disposal.
12. Continue venting until container is down to (near) atmospheric pressure (about 10 psig), then close valve (5).

*Purging time required depends upon many factors which differ in individual systems (such as line diameter, line length, purge rate available, previous use of line, etc.). The diborane user is warned to be sure purge time is more than adequate.

13. Recheck that valves (4) and (6) are closed; then open container valve (2).
14. Open valve (4) to start flow of nitrogen through dip tube into diborane tank. Check pressure rise on container as an indication that dip tube is not plugged.
 - a. If dip tube is not plugged, open valve (5) to release pressure to disposal system. Continue purging with nitrogen until there is no more green flame (probably about 10 to 15 minutes); then close valve (4) and leave valve (5) open.
 - b. If dip tube is plugged, open valve (5) venting container to burn barrel; and try increasing nitrogen pressure. If dip tube remains plugged with 500 psig nitrogen pressure, try maintaining this 500 psi differential for several more hours. If this fails to discharge the plug, proceed as follows:
 - (1) Shutoff nitrogen supply by closing valve (4). Vent line by opening valve (6), then close valve (6). Note that valve (5) is left open.
 - (2) Purge the container by pressure and release.
 - (a) Open nitrogen to container through valve (3). After pressure has built-up, close valve (3) and open valve (5) to vent to burn barrel.

- (b) Repeat pressure and release until there is no green flame and smoke when venting to the burn barrel. This will probably require at least 6 to 10 times.
- (c) Finish with valve (3) closed and valve (5) open.
- (3) Open naphtha* to dip tube.
 - (a) First shutoff nitrogen supply by closing valve (8).
 - (b) Open valve (4) to container and valve (6) to burn barrel.
 - (c) Open naphtha supply valve (10).

DANGER: Do NOT use methanol or isopropanol.
- (4) When naphtha comes out line to the burn barrel, close valve (6).
- (5) Close naphtha supply valve (10), and open nitrogen supply valve (8).
- (6) If plug does not dislodge, try increasing nitrogen pressure.
- (7) If dip tube remains plugged with nitrogen pressure raised to 500 psig, try maintaining this 500 psi differential for several more hours.

*Water-free Naphtha; Pennsylvania Refining Company 180/250°F., or equivalent.

(8) If this fails to discharge the plug, it must be due to some unusual conditions or treatment of the container; and this information would have to be known before deciding what procedure to use.

15. Proceed with naphtha* wash when dip tube is open. First shutoff nitrogen supply by closing valve (8); then open valve (4) to vent nitrogen pressure through the container. Open valve (10) to start naphtha flow through valve (4) into dip tube.

DANGER: Make sure naphtha is added first, NOT isopropanol or methanol.

Normally there is little or no reaction on adding naphtha, but the operator should watch for pressure rise.

16. If there is no evidence of reaction, continue charging naphtha until the container overflows through valve (1) and valve (5) to the burn barrel.
17. After naphtha overflows, close valve (5) and open valve (14) to flush naphtha through line to pressure gauge and out the gauge purge valve (14).
18. Close valve (14) to stop naphtha flow. Shutoff naphtha supply by closing valve (10), and open nitrogen supply through valve (8). Raise nitrogen pressure to 300 psig

*Water-free Naphtha; Pennsylvania Refining Company 180/250°F., or equivalent.

to force naphtha into working elements of pressure gauge for about 5 minutes.

19. Close nitrogen supply valve (8); then vent container by opening valve (5) and valve (4).
20. Leave vented container filled to overflowing with naphtha for a minimum of 24 hours. Under certain conditions an overnight time of 16 hours may be adequate, but only when: (1) the diborane has never been permitted to warm up above -35°C . AND (2) total diborane storage time, full or empty, has been less than six weeks.
21. When the required time has elapsed, remove naphtha from container by opening nitrogen supply valve (8). With valves (4), (2), (1), and (5) still open, the naphtha will be pressured out through the dip tube to the burn barrel*. Open valve (14) to flush gauge line.
22. Continue until naphtha is removed from container, and nitrogen comes out line to burn barrel for one or two minutes. Then close nitrogen supply valve (8).
23. Confirm that valves (4), (2), (1), and (5) are still open; then proceed with isopropanol** wash.

DANGER: Make sure isopropanol is added, NOT methanol

*It may be possible to reuse the naphtha, but this cannot be recommended as a blanket procedure. Used naphtha must be stored in vented containers.

**Anhydrous isopropanol.

24. Carefully start flow of isopropanol into container through the dip tube by partially opening valve (11).
WARNING: Introduce isopropanol VERY SLOWLY at the start.
25. As isopropanol is added, watch for pressure rise; and control rate of addition accordingly. Normally after about 50 gallons has been added, the most violent reaction is completed; and the rate can be increased.
26. Continue charging isopropanol at a safe rate until the container overflows through valve (1) and valve (5) to the burn barrel.
27. After isopropanol overflows, close valve (5) and open valve (14) to flush isopropanol through line to pressure gauge and out the gauge purge valve (14).
28. Close valve (14) to stop isopropanol flow. Shutoff isopropanol supply by closing valve (11), and open nitrogen supply through valve (8). Raise nitrogen pressure to 300 psig to force isopropanol into working elements of pressure gauge for about 5 minutes.
29. Close nitrogen supply valve (8); then vent container by opening valve (5) and valve (4).

30. Leave vented container filled to overflowing with isopropanol for a minimum of 24 hours. Under certain conditions an overnight time of 16 hours may be adequate, but only under the same conditions noted earlier for the naphtha wash (step 20).
31. When the required time has elapsed, remove isopropanol from container by opening nitrogen supply valve (8). With valves (4), (2), (1), and (5) still open, the isopropanol will be pressured out through the dip tube to the burn barrel*. Open valve (14) to flush gauge line.
32. Continue until isopropanol is removed from container, and nitrogen comes out line to burn barrel for one or two minutes. Then close nitrogen supply valve (8).
33. Confirm that valves (4), (2), (1), and (5) are still open; then proceed with methanol wash.
34. Carefully start flow of methanol into container through the dip tube by partially opening valve (12).

DANGER: Introduce methanol VERY SLOWLY at the start.

*It may be possible to reuse the isopropanol, but this cannot be recommended as a blanket procedure. Used isopropanol must be stored in vented containers.

35. As methanol is added, watch for pressure rise; and control rate of addition accordingly. Normally after about 50 gallons has been added, the most violent reaction is completed; and the rate can be increased.
36. Continue charging methanol at a safe rate until the container overflows through valve (1), and valve (5) to the burn barrel.
37. After methanol overflows, close valve (5) and open valve (14) to flush methanol through line to pressure gauge and out the gauge purge valve (14).
38. Close valve (14) to stop methanol flow. Shutoff methanol supply by closing valve (12), and open nitrogen supply through valve (8). Raise nitrogen pressure to 300 psig to force methanol into working elements of pressure gauge for about 5 minutes.
39. Close nitrogen supply valve (8); then vent container by opening valve (5) and valve (4).
40. Leave vented container filled to overflowing with methanol for a minimum of 24 hours. Under certain conditions an overnight time of 16 hours may be adequate, but only under the same conditions noted earlier for the naphtha wash (step 20).

41. When the required time has elapsed, remove methanol from container by opening nitrogen supply valve (8). With valves (4), (2), (1), and (5) still open, the methanol will be pressured out through the dip tube to the burn barrel*. Open valve (14) to flush gauge line.
42. While removing methanol, collect a sample for inspection. Note whether sample is clear and colorless (compare to fresh methanol sample).
43. Continue until methanol is removed from container, and nitrogen comes out line to burn barrel for five to ten minutes. Then close nitrogen supply valve (8).
44. Close facility valves (3), (4), (5), and (6). Close container valves (1), (2), and (14).
45. If sample of wash removed was clear and colorless, proceed to drying. If sample of wash removed was visually contaminated (usually a pale yellow color), repeat methanol wash (steps 33 through 45 inclusive).
46. It is strongly recommended that the container pressure gauge be removed for final cleanout and for calibration check. Reinstall gauge before proceeding to drying of container.

*It may be possible to reuse the methanol, but this cannot be recommended as a blanket procedure. Used methanol must be stored in vented containers.

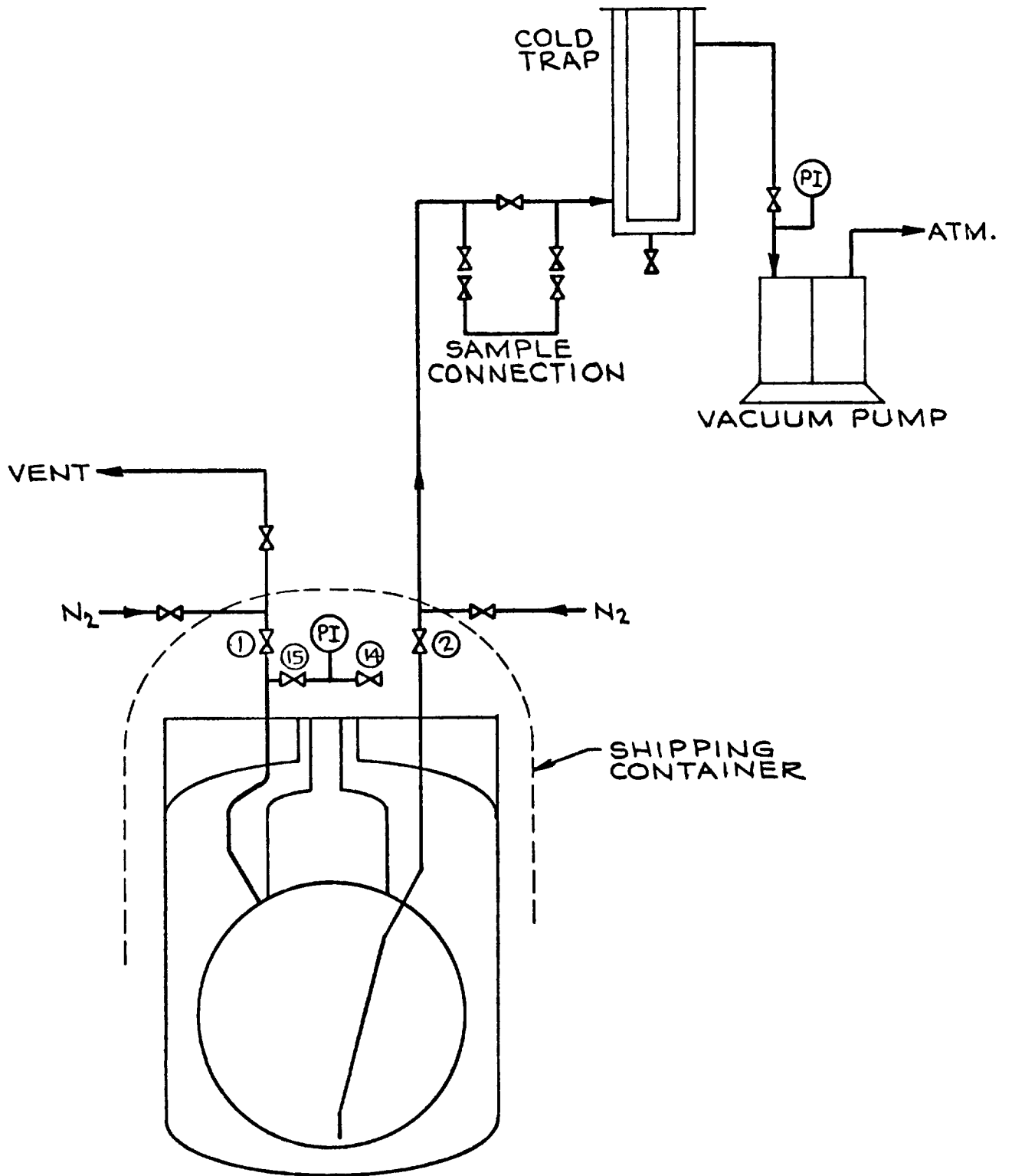
47. Disconnect container from piping system; replace caps on fittings at container valves (1), (2), and (14). Replace valve cover over handle of valve (14).

C. Container Drying

1. Refer to Figure 21 for a Schematic Diagram of Drying Facility. This vacuum drying equipment* may be incorporated as an integral part of facilities for loading and/or unloading diborane from the container.
2. If a suitable scale (over 3000 pound capacity is conveniently available, the container should be weighed prior to drying, but this is not a requirement.
3. Connect the diborane shipping container to a piping system equivalent to that shown in Figure 21.
 - a. Connect container vent/pressurization valve HOV-1 to nitrogen and to vent, via the tee downstream of the valve (1).
 - b. Connect container dip tube valve HOV-2 to the vacuum system and to nitrogen, via the tee downstream of the valve (2).
 - c. Refer back to Figure 20 for location of the above two valves, and to Figure 3 (page 8) for the connection details.

*The same vacuum equipment, if properly designed, may also be used to repump vacuum on the container insulation space, in the event that should become necessary (see Section V.A.2.).

FIGURE 21
SCHEMATIC DIAGRAM OF DRYING FACILITY



4. If the container is located on a scale, recheck weight after connections have been made.
5. This procedure assumes that all valves on Figure 21 are closed at the start, except gauge shutoff valve (15).
6. Open nitrogen to the connection at valve (1) and open nitrogen to the connection at valve (2), but do not open valve (1) and valve (2) yet.
7. Test connections with soap solution to be sure there are no leaking joints.
8. Shutoff nitrogen supply to both valve (1) and valve (2).
9. Cool down the cold trap by means appropriate for the design provided. Figure 21 shows a liquid nitrogen cold trap, which can be used* but is not a requirement; a dry ice-methanol trap or other type might also be used to keep the small amount of methanol out of the vacuum pump.

*A liquid nitrogen cold trap is required for a vacuum facility to repump the insulation space; therefore, such a trap may be used for all vacuum requirements associated with the diborane shipping container.

10. Turn on the vacuum pump, and check that the pump pulls line pressure down to the pump rating before opening to the cold trap. If necessary, change oil in the pump, or perform any other maintenance needed to achieve desired performance of the pump.
11. Open valve between vacuum pump and cold trap; and when that line has pumped down, open valve between cold trap and shipping container. Wait for line to pump down before proceeding.
12. When entire system has pumped down, open container valve (2) gradually, observing pressure decrease on container compound gauge (pressure and vacuum) PI-11. Continue until valve (2) is full open.
13. Drying may now continue virtually unattended, except for whatever attention is required to keep the cold trap cooled down. Depending on the size of the cold trap, it may be necessary to drain the trap to remove methanol.
14. Drying should be continued according to a convenient cycle, in which the vacuum is periodically broken with nitrogen prior to resuming vacuum. If the vacuum system can be operated 24 hours a day, it is recommended that the vacuum be broken with nitrogen at 0800 and at 1600 (for example). No harm is

caused if vacuum is discontinued during overnight or weekend.

15. It is recommended that during at least one overnight, the container be left under vacuum with valve (1) and valve (2) closed to check for possible leakage. Any significant rise may indicate a leak to be repaired during the preliminary checkout prior to loading with diborane.
16. Most of the methanol will be removed in the first 24 to 48 hours, and use of the cold trap could (using a gas ballast vacuum pump) be discontinued at that point if so desired. Scale reading (if available) will help determine that point.
17. Total vacuum time required to dry the container (i.e., remove every trace of methanol) cannot be specified, because there are so many variables (vacuum pump capacity, line sizes of piping system, temperature, frequency of nitrogen purging, etc.).

CAUTION: Do not use heat in any form to accelerate drying; because this will cause the insulation space pressure to rise, making it necessary to repump the insulation space.

Typically 150 to 200 hours of vacuum time may be required to remove all of the methanol.

18. To determine whether the container is dry of methanol, collect a sample using a sample connection similar to that shown in Figure 21.

Procedure should be as follows:

- a. Immerse the sample tube in a bath of dry ice-methanol (or equivalent), and allow at least 30 minutes for cooldown.
- b. Close container valve 2; and break vacuum with nitrogen through container valve 1, allowing pressure to rise to about 5 psig.
- c. Close sample connection bypass valve, and open all four valves to direct flow through sample tube.
- d. With vacuum pump operating as usual, gradually open container valve (2) to pump all gas from the container through the sample tube. Continue to throttle flow with container valve (2), so that total pumdown time is at least 30 minutes.
- e. When container pressure gauge PI-11 shows full vacuum, close all four valves on sample connection; then open sample bypass valve to continue pumping while sample is analyzed. Sample of any condensable material (methanol or water) will be trapped in sample tube for analysis.

19. Analysis of the sample will depend on the type of laboratory equipment available; mass spectrometer or infrared analysis are two possibilities.
20. Depending on the urgency of getting the container dried, samples might be taken daily or every other day after pumping at least 100 hours.
DANGER: Never try to shortcut the drying operation. An explosion may result if diborane is charged into a container with small quantities of methanol.
21. When analyses show no detectable methanol by the recommended sampling procedure, break the vacuum with nitrogen and leave 10 to 15 psig nitrogen pressure on the container with container valves (1) and (2) closed.
22. Shutdown and secure the vacuum system. Disconnect lines from the container and replace caps on fittings at container valves.

DIBORANE SHIPPING CONTAINERDATA RECORD - CLEANOUTReceiving "Empty" Container

Pressure _____ psig

Action Taken _____

Visual Appearance _____

Remarks _____

Container Washout

Container Weights: Gross _____

Tare _____

Net _____

Container Vented _____ (date)

Dip Tube Open _____ (date)

	<u>Date In</u>	<u>Date Out</u>
Naphtha Wash	_____	_____
Isopropanol Wash	_____	_____
Methanol Wash	_____	_____

First Methanol Wash

☐

Clear

☐

Cloudy Yellow

Action Taken _____

Container Washout (Cont)

Maintenance Performed

Pressure Gauge Checked

Other (_____)
_____APPROVED FOR DRYING by _____
OPERATIONS SUPERVISORContainer Drying

Container Weights: Initial _____ lbs.

Final _____ lbs.

Vacuum Times:

Date	Time		Remarks
	Start	Stop	
_____	_____	_____	_____
_____	_____	_____	_____
_____	_____	_____	_____
_____	_____	_____	_____
_____	_____	_____	_____
_____	_____	_____	_____
_____	_____	_____	_____
_____	_____	_____	_____
_____	_____	_____	_____

Samples:

Date	Result
_____	_____
_____	_____
_____	_____

APPROVED DRY (NO METHANOL) by _____
OPERATIONS SUPERVISOR

V PROCEDURE FOR LOADING

WARNING: DIBORANE SAFETY AND HANDLING INFORMATION IN SECTION III OF THIS MANUAL MUST BE READ AND UNDERSTOOD BEFORE PROCEEDING WITH LOADING OF DIBORANE.

Detailed procedures for loading of diborane into the shipping container from a storage facility of necessity depend upon the design of that facility; therefore, these instructions can be complete only in terms of the shipping container itself. The customer must prepare additional procedures for the facility from which the diborane is removed.

Section 4 of DIBORANE HANDBOOK (N70-78384) by M. T. Constantine et al. of the Research Division of Rocketdyne (a Division of North American Rockwell Corporation) prepared for NASA under Contract NAS7-769 contains very complete information on Storage and Handling of diborane. All applicable sections of that compilation should be considered in both facility design and worker education/training.

General Safety and Handling Procedures (From DIBORANE
HANDBOOK, Section 5.4.2)

5.4.2 Safety and Handling Procedures

During any operations involving the handling or transfer of B_2H_6 containers, the safety precautions and handling procedures noted in Sections 6 and 4.5, respectively, should be observed. In general, these procedures include the following:

1. Any person involved in the handling or transfer of B_2H_6 from its container should be thoroughly familiar with the physical, chemical, and engineering characteristics of B_2H_6 .
2. Two operators should be available at all times during any operation involving the handling, transfer, or storage of B_2H_6 , and they should be sufficiently separated so that a mishap would not incapacitate both.
3. All personnel not directly concerned with the handling operation should evacuate the hazard area.
4. Personal safety equipment should be worn during all handling operations. (See Section 6.4.1.2)
5. Water hoses, showers, and other appropriate safety equipment should be available in the immediate vicinity of the handling area. The location and proper function of this equipment should be checked before beginning any operation.
6. Whenever possible, B_2H_6 -containing systems should be remotely controlled.

LOADING

There are two basic methods for loading diborane into this shipping container: (1) External Condenser and (2) Liquid Transfer. In either procedure, diborane must be charged by weight; so a scale capable of weighing 3000 pounds is required. Either method requires that the container be precooled; either to -80°C . with liquid nitrogen, or to -70°C . with dry ice (or liquid nitrogen). In any case, certain preliminary checkout is required prior to precooling and charging diborane.

A. Preliminary Checkout

1. Check condition of the battery, which is power source for the high-temperature alarm and for the level indicator.

a. Battery location is shown in Figure 8 and Figure 13.

b. Battery voltage is checked by activating the voltage indicator (see also page 16).

(1) Selector switch must be in the "ON" position.

(2) Depress voltage indicator pushbutton while reading percentage scale.

(3) Note whether reading is within the red-lined part of the scale.

c. When reading is in the red-lined section, replace battery:

Burgess Battery #302580
27v., 3400 MAH

2. Check insulation space vacuum

- a. Location of sensing element for pressure in insulation space is shown in Figure 12. Specifications for this item (part of the container) are as follows:
 - Type - Hastings DV-6M metal gauge tube
 - Response - .06 second time constant (air release to atmosphere).
 - Warm up time - less than 3 seconds.
 - Stability - .5% 90-140 volt line change.
 - Compensation - ambient temperature and rate of change.
- b. Readout instrument (not part of the container; to be supplied by shipper) should be as follows (or equivalent):
 - Type - Hastings VT-6B cabinet model
 - Range - 0 to 1000 microns of Mercury
 - Power - 115 volt AC/DC less than 1 watt
 - Cable - 8' cable to gauge tube
 - Available with or without switching unit
- c. Container should be at normal temperature (no dry ice or liquid nitrogen) at the time reading is taken.
- d. Record appropriate information on data record.
- e. Pressure must be 50 microns or lower at equilibrium temperature of 50°F. or higher.
- f. If necessary, insulation space must be repumped to a better vacuum using a mechanical pump capable of one micron, a liquid nitrogen cold trap, and a properly designed piping system (large diameter, short lines; free of any leaks).

- (1) Repumping should be continued until pressure is down to at least 25 microns (preferably lower) at ambient temperature of 70°F. or higher.
- (2) Pressure should be monitored for at least one week, with no rise in pressure (loss of vacuum) not attributable to temperature change. Any leaks must be repaired.

g. Record appropriate information on data record.

3. Pressure test diborane system by:

- a. Pressurize with dry nitrogen (or helium) to at least 150 psig on PI-11.
- b. Read exact pressure, and recheck later (one day or longer) for loss of pressure not attributable to temperature change.
- c. Check for leakage at all exposed joints, using soap solution. Particularly, check the threaded joints where the level probe and temperature probe enter the top of the inner tank, at the bottom of the dry ice chamber.*
- d. ALL LEAKS, NO MATTER HOW SMALL, MUST BE STOPPED.
- e. Record appropriate information on data record.

*For convenience in tightening these connections, special wrenches with extension handles may be used:

7/16" Socket Head with 39" T-handle
15/16" Socket Head with 39" T-handle
1-1/16" Open End cut to 4-3/4" length

B. Precooling to -80°C .

DOT Special Permit No. 6522 (First Revision) requires that the container be precooled to below -80°C . with liquid nitrogen. The recommended procedure, used in the certification testing, is as follows:

1. All work in the preceding section (V.A.) must be completed prior to start of precooling.
2. Precooling should not be started until plans have been made for loading with diborane and shipment of the container.
 - a. About 10 percent more than the required amount of diborane should be available for charging on the eighth day after start of precooling.
 - b. Shipment should be scheduled for the ninth day after start of precooling (or for the eighth day if shipment can be arranged the same day as loading).
3. Setup back-pressure regulator (or suitable pressure relief valve) and manual vent (bypass of regulator or relief valve) from the tee downstream of the container vent valve (HOV-1 in Figure 4, connection details in Figure 3). Back pressure control setting is not critical, and may be at any convenient pressure around 5 to 50 psig; its purpose is to prevent condensation of water in the

liquid nitrogen, which could occur in an open-vented container over a period of time.

4. Setup connections between liquid nitrogen source and tee at container dip tube valve (HOV-2 in Figure 4, connection details in Figure 3). The cap may be left on one side of the tee.
5. Open container valves HOV-1 and HOV-2. Open vent valve (bypass of back-pressure regulator), and leave open during the liquid nitrogen charging operation.
6. Transfer the liquid nitrogen from three 50-liter containers (or the equivalent) into the inner tank of the shipping container.
7. Initially most of the liquid nitrogen will be vaporized in cooling the warm container. As the container cools down, rate of gaseous nitrogen evolution will decrease. Shortly after the specified amount of liquid nitrogen has been charged, it should be possible to close the manual vent valve and direct vent through the back-pressure regulator.
8. If the container is located on a scale at this time, liquid nitrogen weight may be checked; net after charging the 150 liters will probably be about 100 to 120 pounds.

9. Liquid nitrogen should be recharged the second or third day, before the initial charge is all vaporized. Another three 50-liter containers (or equivalent) should be charged.
10. Upon charging the 150 liters in step 9 above, weight will probably increase by about 200 pounds, since the container will be colder than during the initial charge.
11. While the container is cold, preferably on the sixth or seventh day, another leak test should be performed. With dip tube valve HOV-2 closed, pressurize through vent/pressurization valve HOV-1 (with back-pressure regulator valved off or set high enough) to 150 psig with dry nitrogen. Test as described in Preliminary Checkout Section, V.A.3.c. and d. Use methanol in the soap solution used for testing for leakage on connections at the bottom of the dry ice chamber.
12. On the seventh day the excess liquid nitrogen should be removed (or on the day before charging diborane, whether the seventh day or longer). Using nitrogen pressure on the container, the excess liquid nitrogen is pressurized out through the dip tube until the liquid nitrogen level is below the bottom of the dip tube.
13. After standing overnight (16 hours), the precooling operation is completed ready for charging diborane. The back-pressure regulator may be removed, after closing

the manual valve (HOV-1) to maintain a positive pressure of nitrogen on the container until ready to add diborane.

14. Record appropriate information on data record.

C. Precooling to -70°C .

DOT Special Permit No. 6522 (Second Revision) permits shipment of a container precooled to -70°C . (instead of -80°C .), provided delivery is made within the time required by the permit. The recommended procedure is written for use of dry ice, since liquid nitrogen would normally be used only for the -80°C . precooling already discussed.

1. All Preliminary Checkout (Section V.A.) must be completed prior to start of precooling.
2. Precooling should not be started until plans have been made for loading with diborane and shipment of the container.
 - a. About 10 percent more than the required amount of diborane should be available for charging upon completion of precooling.
 - b. Shipment should be pre-scheduled for the day on which charging of the container is completed.
3. Before adding dry ice to the dry ice chamber, check to be sure there is a positive nitrogen (or helium)

pressure of about 25 to 50 psig on the inner tank.
ALWAYS MAINTAIN A POSITIVE INERT GAS PRESSURE IN A
CLEAN CONTAINER PRIOR TO CHARGING.

4. The quantity of diborane charged and shipped must be established by weight; therefore, the constantly-changing dry ice weight must be eliminated as a variable when recording the initial tare weight and the final gross weight of the container.
 - a. Initial tare weight, or empty weight, may be recorded prior to start of dry ice precooling. It is also important that tare weight be measured at (or converted to) atmospheric pressure; for example, 100 psig nitrogen will add about 6.5 pounds to the tare weight.
 - b. Because rate of dry ice loss is only approximately predictable, and less so while diborane is being charged, it is recommended that dry ice be charged into a bag inside the dry ice chamber; so that when down to about 10 or 20 pounds of dry ice, the bag containing the dry ice may be removed to enable obtaining an accurate weight.
5. Having taken into account the above conditions, proceed to charge dry ice into the dry ice chamber as follows:

- a. Record weight of container before adding any dry ice. Note any conditions which may affect weight (internal pressure, any lines connected, side panel on or off scale, etc.), and record other container instrument readings.
 - b. Breakup the dry ice into small lumps, either with an ice crushing machine or manually.
 - c. To add dry ice, remove the thermal plug (Figure 1) by loosening the four 1/4-inch slotted hex head machine screws, lift out the plug by the handle, and drop in the dry ice through the neck into a bag in the chamber below.
 - d. To achieve a high packing density, dry ice may be compacted by tamping as with the end of a wood two-by-four; using caution to prevent damage to instruments at the bottom of the dry ice chamber.
 - e. Pack dry ice into the chamber reasonably full, with allowance for insertion of the thermal plug.
 - f. Reinstall thermal plug, and fasten securely to the container.
 - g. Check and record gross weight; calculate and record net weight of dry ice added.
6. Record container gross weight at least two times a day initially; check rate of dry ice loss and drop in reading on temperature indicator. Make sure a positive pressure is maintained on the inner tank during cooldown.

7. Rate of dry ice loss will be quite high in the beginning, because the container will be warm at the start; but after a few days, dry ice loss rate should drop to about 20 pounds per day. Charging of diborane may begin after the container has been precooled to $-70^{\circ}\text{C}.$; however, it may be advisable to allow about one day of continued cooling after indicated temperature reaches $-70^{\circ}\text{C}.$
8. When precooling is completed, remove any residual dry ice to recheck weight with connections made ready for charging. Add dry nitrogen pressure to container inner tank to about 150 psig and test for leaks as described in Preliminary Checkout Section, V.A.3.c. and d. Use methanol in the soap solution used for testing for leakage on connections at the bottom of the dry ice chamber.
9. Maintain supply of dry ice inside bag in dry ice chamber until ready to feed diborane; this can also be maintained during diborane addition except at times when checking weights. Maintain positive pressure of nitrogen on inner tank until vented for diborane charge.
10. Record appropriate information on data record.

D. Charging Diborane - External Condenser

DOT Special Permit No. 6522 requires that the container be filled by weight; therefore, a scale with capacity of

at least 3000 pounds is required.

1. All work in the preceding sections (V.B. or V.C.) must be completed prior to start of diborane charge. In particular:

a. About 10 percent more than the required amount of diborane should be available.

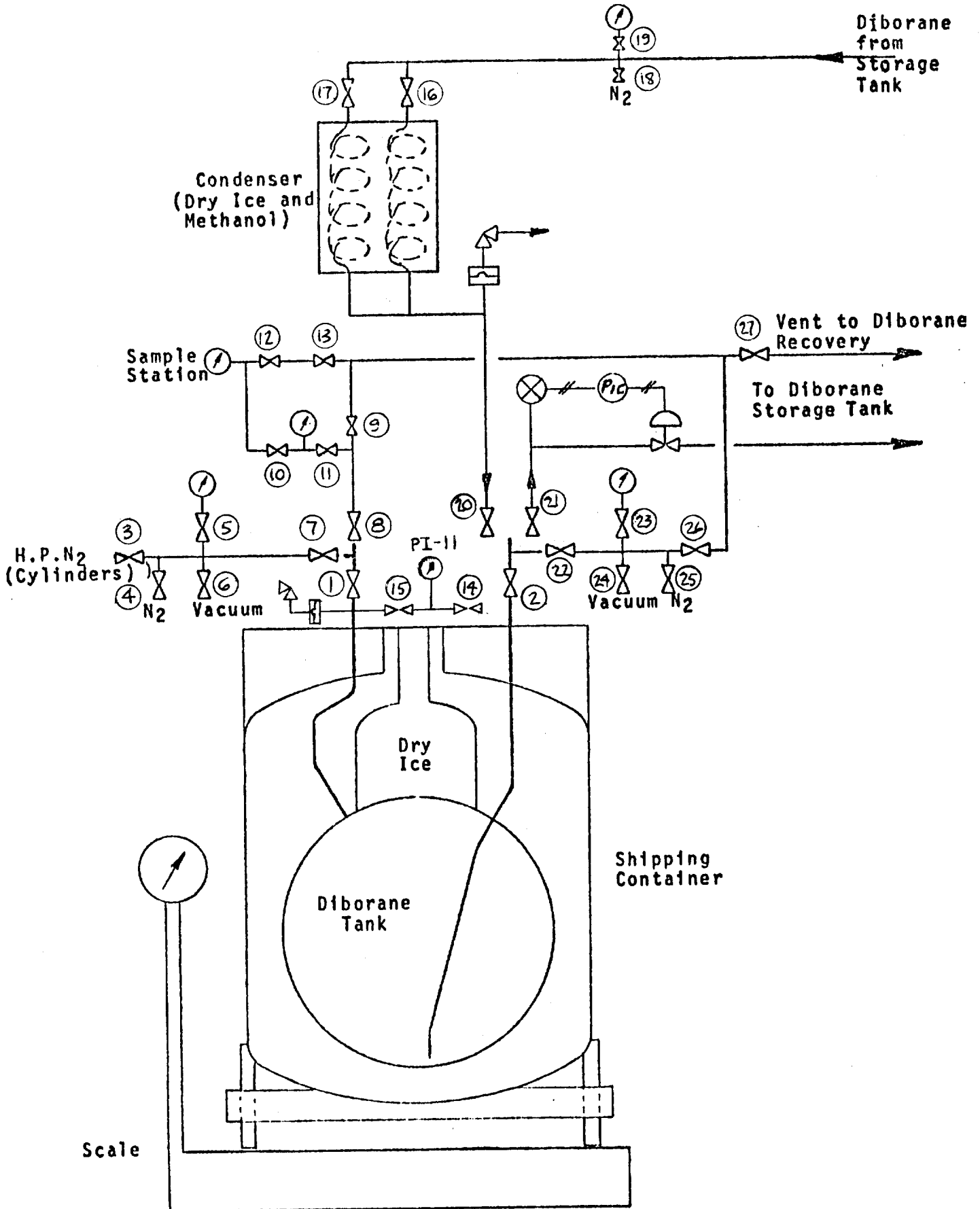
b. For loading at -80°C . after liquid nitrogen pre-cooling, the excess liquid nitrogen should be removed 16 to 24 hours prior to start of diborane charging.

2. Refer to figure 22 for a Schematic Diagram of Diborane Loading Facility with External Condenser. This is essentially the facility used by Callery Chemical Company in loading the container for certification tests.

3. All valves shown in Figure 22 are to be closed at the start; except gauge purge valves (5), (15), and (19).

FIGURE 22

SCHEMATIC DIAGRAM of DIBORANE LOADING FACILITY
with EXTERNAL CONDENSER



4. This procedure presumes that the transfer lines are clean, and therefore it is only necessary to purge lines to displace the air which may be present if not totally sealed under inert gas blanket. Furthermore this procedure presumes that the entire system has been properly maintained and tested to be free of any leaks. THE OPERATOR MUST NOT PRESUME, HE MUST KNOW THAT THE SYSTEM IS CLEAN AND FREE OF LEAKS.
5. Position and secure the container on the scale. Read and record weight.
6. Remove side panel and set aside for reassembly after loading has been completed. This exposes about half of the circumference of the container down about one foot from the top, providing access to both the valve quadrant and the instrument quadrant.
7. It is desirable, but not necessary, to connect the downstream side of the container pressure relief valve into an emergency vent header. Thus if the rupture disk and relief valve were opened by overpressure, the diborane could be piped to a more remote point of release. Such piping should be of an inside diameter at least as large as 3/4-inch Schedule 40 pipe, and should contain a minimum number of elbows (use 45-degree instead of 90-degree elbows). This line should contain NO manual shutoff valves, and should discharge open to

the atmosphere. The relief valve outlet is 3/4-inch female NPT, so it will accept a 3/4-inch male NPT connection. Flexible connection should be made to prevent placing any strain on the relief valve and/or its inlet piping.

8. Identify the two valves where connections are to be made. Facing into the valve compartment, the vent valve (1) is on the left; and its tubing line connects to the pressure relief devices. The liquid inlet valve (2) is on the right, nearer to the partition between the valve quadrant and the instrument quadrant.
9. Remove caps from tees where connections are to be made. Keep caps for reinstallation on the container in preparation for shipment.
10. Confirm that there is still a positive pressure of nitrogen on the container by checking container pressure gauge PI-11.
11. Connect lines from loading facility to shipping container, as shown in Figure 22. These lines should be flexible to prevent placing any strain on container valves and piping. MAKE ABSOLUTELY SURE LINES ARE CONNECTED TO THE CORRECT FITTING (see item 8 above). These lines may be connected loosely for initial purging.

12. In Figure 22, the container dip tube valve (2) may be connected either to load or unload the diborane. For this procedure, connect valve (2) to valve (20), which is the drain from the external condenser.

13. Purge transfer line by flowing nitrogen (or helium) through, as follows:

- a. Open nitrogen supply valve (18) through one condenser coil valve (16) and through valves (20), (22), (26), and (27) to vent; purge for about 3 to 5 minutes.* Loosen the connections to the tee at valve (2) during this purge; then tighten both fittings securely.
- b. After the required time, close valve (16) and open the other condenser coil valve (17); and purge for an equal time.

NOTE: This procedure assumes that the line from the diborane storage tank (not shown) to valves (16) and (17) has already been purged.

- c. Close valve (26) at the end of the purge time; and reopen valve (16) to the other condenser coil.
- d. Open nitrogen supply valve (4) through valves (7), (8), (9), and (27) to vent; purge for about 3 to

*Purging time required depends upon many factors which differ in individual systems (such as line diameter, line length, purge rate available, previous use of line, etc.). The diborane user is warned to be sure purge time is more than adequate.

5 minutes*. Loosen the connections to the tee at valve (1) during this purge; then tighten both fittings securely.

- e. After the required time, close valve (9) and open sample station valve (11), (10), (12), and (13) to purge sample tube for an equal time.
- f. Close valves (27), (13), (12), (10), and (11) at the end of the purge time; and reopen valves (9) and (26).
- g. Leave nitrogen supply valve (4) open and check that nitrogen supply valve (18) is still open. This permits pressure buildup to a predetermined level for leak testing of connections and transfer lines.

14. When system is shown to be free of any leaks, proceed to final purging by pressure and release; according to the following steps:

- a. Close nitrogen supply valves (4) and (18), and vent through valve (27). If available, vacuum valves (6) and/or (24) may be opened to evacuate piping.
- b. Close vent valve (27) and/or vacuum valves (6) and (24), and repressure with nitrogen through valve (4) or valve (18).

*Purging time required depends upon many factors which differ in individual systems (such as line diameter, line length, purge rate available, previous use of line, etc.) The diborane user is warned to be sure purge time is more than adequate.

- c. Repeat two more times, ending by venting through valve (27).
15. Procedure for the diborane transfer itself will depend to some extent on the design and operating conditions of the storage facility, including the vent system and diborane recovery capabilities. This procedure assumes that high purity gaseous diborane is supplied from the storage tank (where it may be stored as a liquid).
16. Basically the transfer consists of condensing gaseous diborane under a pressure of about 100 psig (or as required by facility design), with the liquid diborane drained from condenser(s) into the dip tube of the shipping container. Some venting of the shipping container will probably be necessary, depending on temperatures, amount of non-condensable inert gases, etc.
17. Check and record initial weight of container after all connections have been made and just before opening any valves in the transfer line. Dry ice should not be in the dry ice chamber unless it is inside a bag which can be removed to obtain accurate weights. Be certain consistent tare weight is used (if side panel is on the scale, leave it there for both "before" and "after" weights; compare "before" and "after" weights where both are connected to facility piping or where both are disconnected from facility piping).

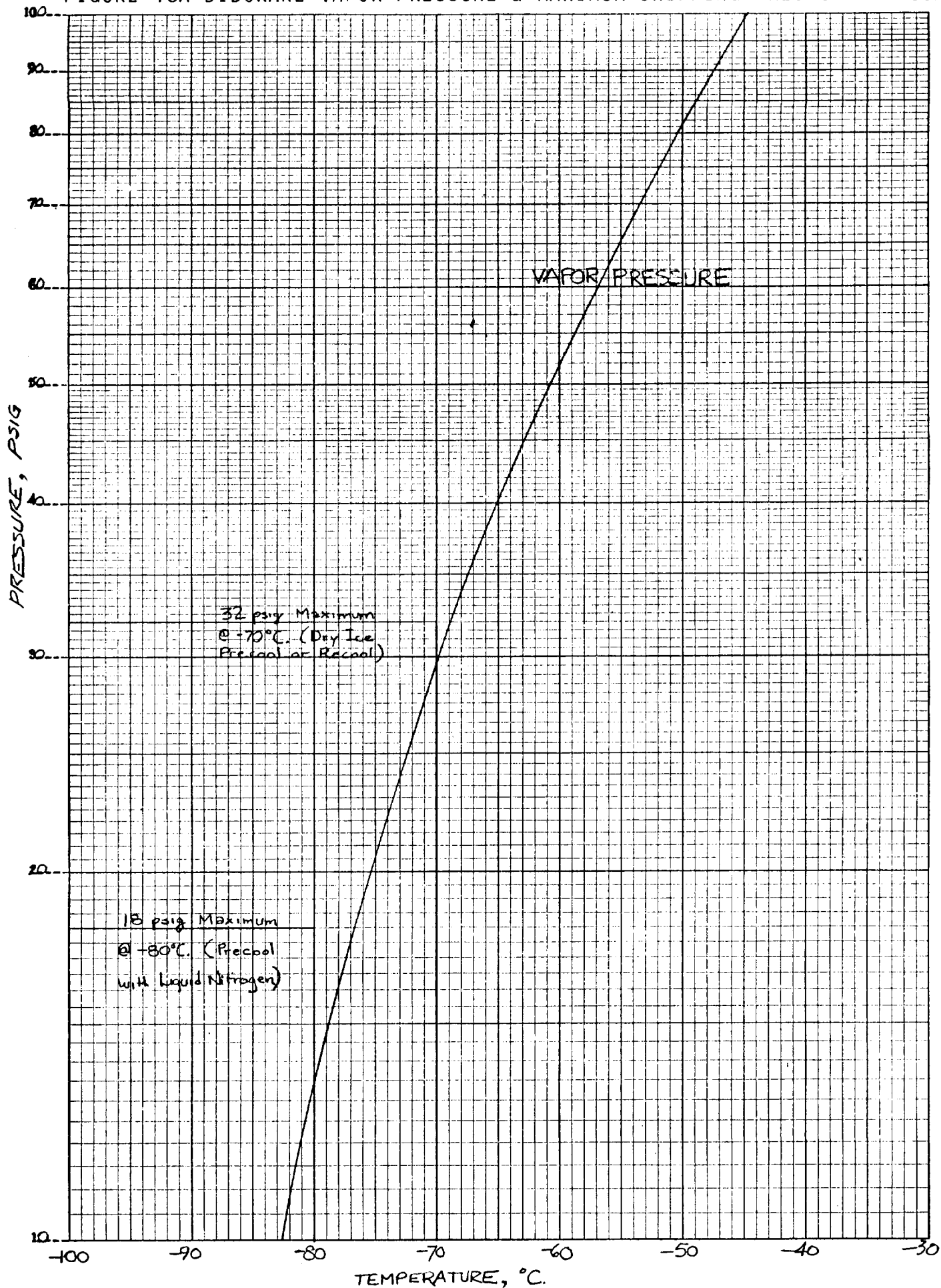
18. With transfer line having been purged as described, and with container confirmed to have positive pressure of nitrogen, proceed* to open container vent valve (1) and dip tube valve (2).
19. After venting shipping container to atmospheric (vent line) pressure, close vent valves (8), (22), and (26). Subsequent venting depends on the facility, and will be discussed in steps at the end of diborane transfer.
20. Condenser should be cooled with dry ice and methanol (or equivalent) a suitable time in advance. Close valve (20) between condenser and shipping container when ready to start transfer.
21. Open storage tank outlet valve (not shown) to begin transfer of diborane; leave full open to condenser during transfer.
22. Subsequent control will depend on the rate at which diborane will condense in the facility used. After liquid diborane collects in the condenser, begin draining by partly opening valve (20); but restrict drainage to a rate which will maintain a liquid seal.

*It is strongly recommended that a sample of the inert gas in the container be collected while venting; for analysis to confirm it is 100% nitrogen, with no trace of methanol or other foreign material.

23. Monitor scale reading to check on rate of transfer, and as a guide to controlling drain rate of liquid diborane from the condenser with valve (20).
24. Monitor shipping container pressure; which should rise very slowly if the container was properly precooled, if adequate dry ice is used in the condenser, and if inert gas transferred with the diborane is negligible.
25. If there should be enough inert gas fed with the diborane that container pressure equalizes with storage tank pressure, it will be necessary to do an intermediate venting operation. Procedure is the same as for the final venting operation outlined below.
26. Continue transfer until the required net weight of diborane is in the container. Note that if pressure is higher than 18 psig (for -80°C. shipment) or 32 psig (for -70°C. shipment), the container must be vented to that pressure or below; and in doing so, some diborane will be "lost" to the vent system. In such cases, the container should be charged with an appropriate excess of diborane, and brought to an exact desired net weight by venting the required amount.

27. Venting is recommended as the last step of loading in any case, as this effectively "tops" non-condensables to assure highest purity. A sample may be collected at this time for analysis, if required.
28. If venting is unsuccessful in reducing pressure, it is probably because temperature is too high; which may be due to insufficient precooling or to charging diborane at too high a temperature (see Figure 18A). If pressure remains above 32 psig, additional cooling with dry ice is required (see also Section XI). If pressure remains above 18 psig, it would be best to ship under the conditions (shipping time limits) of a container precooled and charged for $-70^{\circ}\text{C}.$, which allows up to 32 psig.
29. In accordance with the above guidelines, close valve (2) to complete the transfer; and reopen valve (20) to full open. CAUTION: Do not close any other valves between the shipping container valve (2) and the diborane storage tank yet.
30. Vent through container valve (1) and line valves (8), (9), and (27) to weight and pressure specifications. Final venting may be directed through sample tube by closing valve (9) and opening valves (11), (10), (12), and (13). Then after closing the latter four valves, the tube with sample trapped between valves (10), and (12) may be removed for analysis of diborane purity.

FIGURE 18A DIBORANE VAPOR PRESSURE & MAXIMUM SHIPPING PRESSURE - 85A -



31. In the event venting reduces weight below the minimum required for shipment while pressure is still above the maximum (18 psig for $-80^{\circ}\text{C}.$ or 32 psig for $-70^{\circ}\text{C}.$), it will be necessary to recharge more diborane after completing the first venting; then venting (or topping) should be repeated after the second diborane charge. When venting is completed, close container valve (1).
32. If facility design requires venting an appreciable quantity of diborane, there should be a compressor/condenser system, an absorber/desorber system, or some other suitable means to recover the diborane. For vent streams containing smaller amounts of diborane, a flare or scrubber might be considered for disposal.
33. Container is now closed, containing the desired net weight of diborane at or below the maximum pressure (18 psig at $-80^{\circ}\text{C}.$ or 32 psig at $-70^{\circ}\text{C}.$); and loading system is ready for purging.
34. Open vent valve (27) to diborane recovery system, and open valves (22) and (26) between condensers and vent valve (27). Close main shutoff valve (not shown) at diborane storage tank. Valves (8) and (9) may also be left open during this time to vent the entire system to the diborane recovery system.
35. When system is completely vented down to atmospheric (vent line) pressure, proceed with purging. The pro-

cedure given below covers purging the system shown in Figure 22. If necessary to purge the line all the way to the storage tank, nitrogen may be admitted there instead of through valve (18).

- a. Open nitrogen supply valve (18) through both condensers initially, valves (16) and (17); and through valves (20), (22), (26), and (27) to vent.

- b. Purge for about 3 to 5 minutes* in this manner; then close valve (16) and purge through valve (17) through one condenser coil for about 3 to 5 minutes*; then close valve (17) and open valve (16) to purge through the other condenser coil for about 3 to 5 minutes*.
- c. Close valve (20) from condenser, valve (16) condenser inlet, and nitrogen supply valve (18). When pressure has vented off, close vent valve (26).
- d. Open nitrogen supply valve (4) and purge through valves (7), (8), (9), and (27) to vent for about 3 to 5 minutes*.
- e. If sample tube is still on and not used, purge that section by closing valve (9) and opening valve (11), (10), (12), and (13).
- f. Close nitrogen supply valve (4); and after pressure vents off, close valves (9), (10), (11), (12), (13) and (8).

36. Complete final purging by pressure and release, as follows:

- a. Open nitrogen valve (4) to fully pressurize; then close valve (4) and evacuate through valve (6) [if vacuum is not available, substitute vent valve(s)].

*Purging time required depends upon many factors which differ in individual systems (such as line diameter, line length, purge rate available, previous use of line, etc.). The diborane user is warned to be sure purge time is more than adequate.

- b. Open nitrogen valve (25) to fully pressurize; then close valve (25) and evacuate through valve (24), [if vacuum is not available substitute vent valve(s)].
 - c. Repeat (a) and (b) above two more times, leaving system vented with all valves closed.
37. Carefully disconnect lines from shipping container, just cracking loose each fitting first before loosening and removing.
 38. Replace the four caps on the two tees on the shipping container valves (1) and (2), and tighten.
 39. Disconnect piping that may have been connected to container relief valve (after any necessary purging, depending where this had been connected into).
 40. Record activity on shipping container data sheet.
 41. Side panel may be installed at this time if container is to be moved, or may be left off until preparing container for shipment.
 42. Container must then be reweighed to confirm correct net weight of diborane; be sure side panel is also on scale, and that any necessary corrections are made for dry ice in the ice chamber. Container tare weight must be stenciled on container and recorded on manual copy with the container.

43. Notify all personnel concerned that the transfer operation is completed.

E. Charging Diborane - Liquid Transfer

DOT Special Permit No. 6522 requires that the container be filled by weight; therefore, a scale with capacity of at least 3000 pounds is required.

1. All work in preceding Sections V.B. or V.C. must be completed prior to start of diborane charge. In particular:
 - a. About 10 percent more than the required amount of diborane should be available.
 - b. For loading at -80°C . after liquid nitrogen pre-cooling, the excess liquid nitrogen should be removed 16 to 24 hours prior to start of diborane charging.
2. Refer to Figure 23 for a Schematic Diagram of Diborane Loading Facility for Liquid Transfer. This is presumed to be a typical loading facility, but those who use this procedure are cautioned to consider the requirements of their specific facility design.
3. All valves shown in Figure 23 are presumed to be closed at the start.

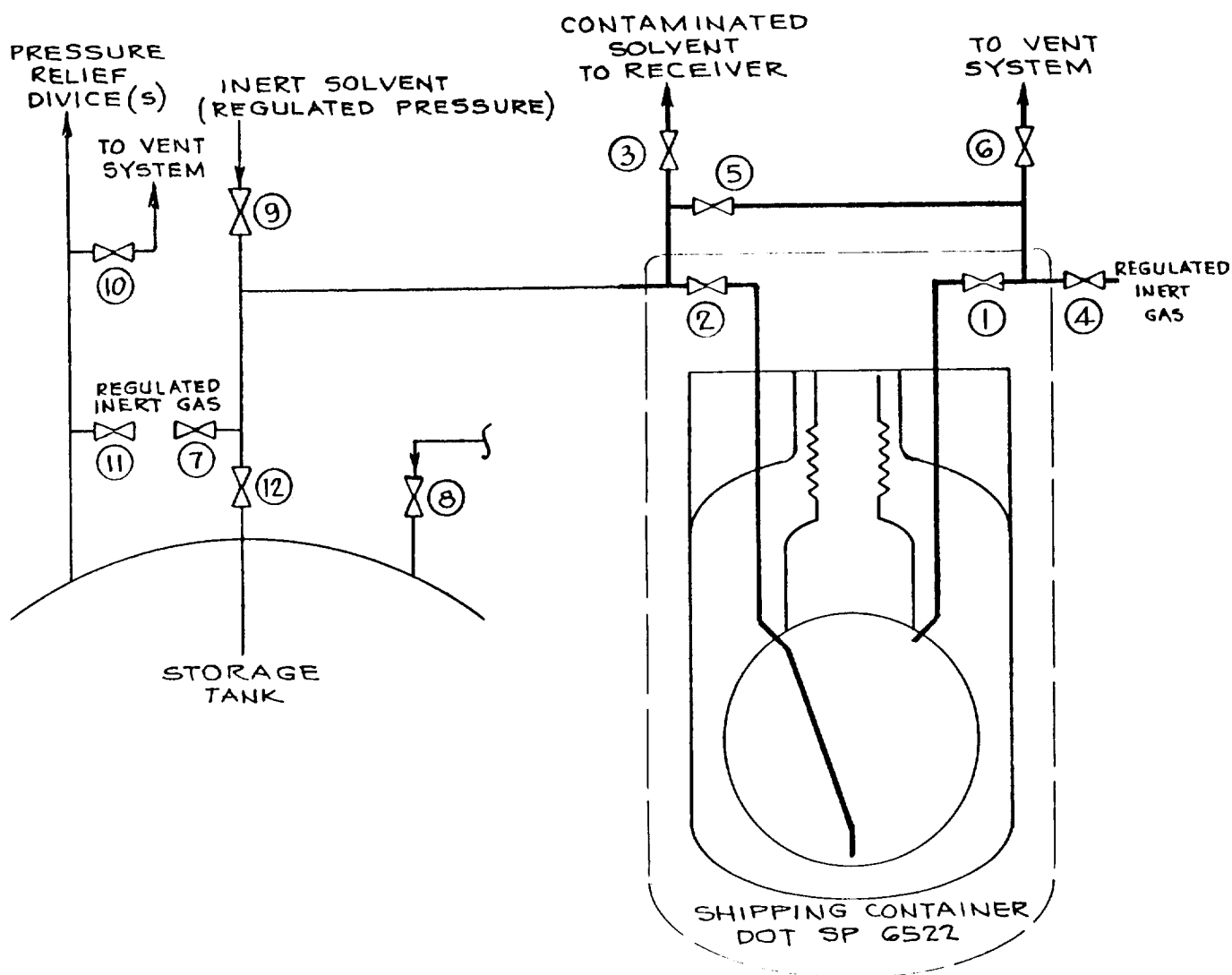
43. Notify all personnel concerned that the transfer operation is completed.

D. Charging Diborane - Liquid Transfer

DOT Special Permit No. 6522 (First Revision) requires that the container be filled by weight; therefore, a scale with capacity of at least 3000 pounds is required.

1. All work in the preceding section (V.B.) must be completed prior to start of diborane charge. In particular:
 - a. About 10 percent more than the required amount of diborane should be available.
 - b. Excess liquid nitrogen should be removed 16 to 24 hours prior to start of diborane charging.
2. Refer to Figure 23 for a Schematic Diagram of Diborane Loading Facility for Liquid Transfer. This is presumed to be a typical loading facility, but those who use this procedure are cautioned to consider the requirements of their specific facility design.
3. All valves shown in Figure 23 are presumed to be closed at the start.

FIGURE 23

SCHEMATIC DIAGRAM OF DIBORANE LOADING FACILITY
FOR LIQUID TRANSFER

- (1) Container Valve - Pressurization or Vent
- (2) Container Valve - Liquid Outlet
- (3) Solvent to waste receiver
- (4) Pressurizing Gas Supply
- (5) Container Bypass Valve
- (6) Container and Transfer Line Vent
- (7) Purge Gas Supply
- (8) Storage Tank Inlet Shutoff
- (9) Inert Solvent Supply
- (10) Storage Tank Vent
- (11) Pressurization Gas Supply
- (12) Storage Tank Outlet Shutoff

4. This procedure presumes that the transfer lines are clean, and therefore it is only necessary to purge lines to displace the air which may be present if not totally sealed under inert gas blanket. Furthermore this procedure presumes that the entire system has been properly maintained and tested to be free of any leaks. THE OPERATOR MUST NOT PRESUME, HE MUST KNOW THAT THE SYSTEM IS CLEAN AND FREE OF LEAKS.
5. Position and secure the container on the scale. Read and record weight.
6. Remove side panel and set aside for reassembly after loading has been completed. This exposes about half of the circumference of the container down about one foot from the top, providing access to both the valve quadrant and the instrument quadrant.
7. It is desirable, but not necessary, to connect the downstream side of the container pressure relief valve into an emergency vent header. Thus if the rupture disk and relief valve were opened by over-pressure, the diborane could be piped to a more remote point of release. Such piping should be of an inside diameter at least as large as 3/4-inch Schedule 40 pipe, and should contain a minimum number of elbows (use 45-degree instead of 90-degree elbows). This

line should contain NO manual shutoff valves, and should discharge open to the atmosphere. The relief valve outlet is 3/4-inch female NPT, so it will accept a 3/4-inch male NPT connection. Flexible connection should be made to prevent placing any strain on the relief valve and/or its inlet piping.

8. Identify the two valves where connections are to be made. Facing into the valve compartment, the vent valve (1) is on the left; and its tubing line connects to the pressure relief devices. The liquid inlet valve (2) is on the right, nearer to the partition between the valve quadrant and the instrument quadrant.
9. Remove caps from tees where connections are to be made. Keep caps for reinstallation on the container in preparation for shipment.
10. Confirm that there is still a positive pressure of nitrogen on the container by checking container pressure gauge PI-11.
11. Connect lines from loading facility to shipping container, as shown in Figure 23. These lines should be flexible to prevent placing any strain on container valves and piping. MAKE ABSOLUTELY SURE LINES ARE CONNECTED TO THE CORRECT FITTING (see item 8 above). These lines may be connected loosely for initial purging.

12. Purge transfer line by flowing inert gas through, as follows:

- a. Open valves (4) and (6), and purge for 3 to 5 minutes*; then close valves (6) and (4).
- b. Open valves (7) and (3), and purge for 3 to 5 minutes*; then close valve (3), and leave valve (7) open.
- c. Open valves (5) and (6), and purge for 3 to 5 minutes*. Loosen the connections to the tees at valve (1) and valve (2) during this purge; then tighten all four fittings securely.
- d. Close valve (6), and leave valve (5) and valve (7) open. This will permit pressure buildup to a predetermined level for leak testing of the connections and transfer line.

13. When system is shown to be free of any leaks, proceed to final purging by pressure and release; according to the following steps:

- a. Close valve (7), and open valve (6) to vent; then close valve (6).
- b. Repeat two more times: open valve (7) until pressure builds up; then close valve (7) and open valve (6) until vented; then close valve (6).

*Purging time required depends upon many factors which differ in individual systems (such as line diameter, line length, purge rate available, previous use of line, etc.). The diborane user is warned to be sure purge time is more than adequate.

c. Close valve (5). All system valves should now be closed again, with system containing dry inert gas.

14. Procedure for the diborane transfer itself will depend to a large extent on the design and operating conditions of the storage facility, including the vent system and diborane recovery capabilities. This procedure requires that liquid diborane be charged to the container at a temperature equal to or below the required shipping temperature (either $-80^{\circ}\text{C}.$ or $-70^{\circ}\text{C}.$).
15. Basically the transfer consists of using storage tank pressure (diborane vapor pressure plus necessary nitrogen or helium pressurization) to force liquid diborane out through the storage tank dip tube through the transfer line into the dip tube of the shipping container. Some venting of the shipping container will be necessary, depending on temperatures, amount of dissolved inert gas, etc.
16. Check and record initial weight of container after all connections have been made and just before opening any valves in the transfer line. Dry ice should not be in the dry ice chamber unless it is inside a bag which can be removed to obtain accurate weights. Be certain consistent tare weight is used (if side panel is on the scale, leave it there for both "before" and "after" weights; compare "before" and "after" weights where both are connected to facility piping or where both are disconnected from facility piping).

17. With transfer line having been purged as described, and with container confirmed to have positive pressure of nitrogen, proceed* to open container vent valve (1) and dip tube valve (2).
18. Open valve (6) to vent shipping container and transfer line to (near) atmospheric pressure (vent line back-pressure, depending on facility design).
19. Subsequent venting depends on the facility, and will be discussed in steps at the end of diborane transfer.
20. Open storage tank outlet valve (12) to begin transfer of liquid diborane.
21. Monitor scale reading to check on rate of transfer. Increased storage tank pressure (within safe limits for the facility) may be used for faster transfer. Container dip tube (liquid inlet) valve (2) may be partially closed to reduce transfer rate.
22. Monitor shipping container pressure; which should rise very slowly if the container was properly precooled, if the diborane charge is at the required temperature (lower than -70 or -80°C.), and if inert gas transferred with the diborane is negligible.

*It is strongly recommended that a sample of the inert gas in the container be collected while venting; for analysis to confirm it is 100% nitrogen, with no trace of methanol or other foreign material.

23. Continue transfer until the required net weight of diborane is in the container. Note that if pressure is higher than 18 psig (for -80°C. shipment) or 32 psig (for -70°C. shipment), the container must be vented to that pressure or below; and in doing so, some diborane will be "lost" to the vent system. In such cases, the container should be charged with an appropriate excess of diborane, and brought to an exact desired net weight by venting the required amount.
24. Venting is recommended as the last step of loading in any case, as this effectively "tops" non-condensables to assure highest purity. A sample may be collected at this time for analysis, if required.
- 24A. If venting is unsuccessful in reducing pressure, it is probably because temperature is too high; which may be due to insufficient precooling or to charging diborane at too high a temperature (see Figure 18A). If pressure remains above 32 psig, additional cooling with dry ice is required (see also Section XI). If pressure remains above 18 psig, it would be best to ship under the conditions (shipping time limits) of a container precooled and charged for -70°C., which allows up to 32 psig.
25. In accordance with the above guidelines, close valve (2) to complete the transfer of liquid diborane; then vent through valves (1) and (6) to weight and pressure

specifications, and close valve (1) and (6).

- 25A. In the event venting reduces weight below the minimum required for shipment while pressure is still above the maximum (18 psig for -80°C . or 32 psig for -70°C), it will be necessary to recharge more diborane after completing the first venting; then venting (or topping) should be repeated after the second diborane charge. When venting is completed, close container valve (1).
26. If facility design requires venting an appreciable quantity of diborane, there should be a compressor/condenser system, an absorber/desorber system, or some other suitable means to recover the diborane. For vent streams containing smaller amounts of diborane, a flare or scrubber might be considered for disposal.
27. Container is now closed, containing the desired net weight of diborane under a pressure 18 psig or less; and loading system is ready for purging.

28. Open inert gas purge valve (4) and container bypass valve (5), and purge transfer lines into storage tank for about 5 minutes*. It may be necessary to vent the storage tank to maintain flow of the inert gas purge (this procedure cannot be specific in terms of a storage system whose design is unknown).
29. Close inert gas purge supply valve (4), bypass valve (5), and storage tank outlet valve (12). Immediately open the transfer line to a vent, either via valve (6) to the vent system or via valve (3) to a vented solvent receiver. This will protect against the circumstance where some liquid may have been heldup in the line.
- NOTE: Procedure can be varied at this point. This procedure will be written for use of an inert solvent such as kerosene (water-free). A thorough purging with inert gas might be substituted if properly planned and executed.
30. If valve (6) had been opened to vent system, close valve (6) and open valve (3) to vented receiver for contaminated solvent. Open inert dry solvent supply valve (9) to purge solvent through transfer line for 3 to 5 minutes*.

*Purging time required depends upon many factors which differ in individual systems (such as line diameter, line length, purge rate available, previous use of line, etc.). The diborane user is warned to be sure purge time is more than adequate.

31. Close solvent supply valve (9) and open inert gas purge valve (7) to purge solvent from line into receiver for 3 to 5 minutes*.
32. Close valve (7) and valve (3).
33. Open inert gas supply valve (4) and open vent valve (6) to purge vent line for 3 to 5 minutes*; then close valve (6) and valve (4).
34. Carefully disconnect lines from shipping container, just cracking loose each fitting first before loosening and removing.
35. Replace the four caps on the two tees on the shipping container valves (1) and (2), and tighten.
36. Disconnect piping that may have been connected to container relief valve (after any necessary purging, depending where this had been connected into).
37. Record activity on shipping container data sheet.
38. Side panel may be installed at this time if container is to be moved, or may be left off until preparing container for shipment.

*Purging time required depends upon many factors which differ in individual systems (such as line diameter, line length, purge rate available, previous use of line, etc.). The diborane user is warned to be sure purge time is more than adequate.

39. Container must then be reweighed to confirm correct net weight of diborane; be sure side panel is also on scale, and that any necessary corrections are made for dry ice in the ice chamber. Container tare weight must be stenciled on container and recorded on manual copy with the container.
40. Notify all personnel concerned that the transfer operation is completed.
41. Procedures for disposal of contaminated solvent are beyond the scope of this manual. The user should have developed this and other facilities/procedures in advance, using the previously referenced DIBORANE HANDBOOK where necessary for guidance.

F. Preparation for Shipment

Proceed with shipment preparation in accordance with the requirements of Section VI following.

1. Confirm that HOV-1 container vent valve is closed with both caps securely fastened to tee fitting.
2. Confirm that HOV-2 container dip tube valve is closed with both caps securely fastened to tee fitting.
3. Confirm that HOV-14 container pressure gauge purge valve is closed with cap securely fastened to end fitting.

4. Confirm that HOV-15 container pressure gauge shutoff valve is open.
5. Turn instrument switch to the "ON" position.
6. Check that correct tare weight is stenciled on container and entered on data records.
7. Check that "DOT SP 6522" is stenciled (minimum 2-inch height) on container.
8. Check that container is labeled in accordance with DOT requirements (refer to Manual Section VI).
9. Check that other required labels are installed (refer to Manual Section VI, Figure 24).
10. Add dry ice in preparation for shipment as follows:
NOTE: DOT Special Permit No. 6522 requires that the net weight of dry ice in the ice chamber be at least 108 pounds on the date of shipment.
 - a. Record weight of container before adding any dry ice, along with other container instrument readings.
 - b. Breakup the dry ice into small lumps, either with an ice crushing machine or manually.

- c. To add dry ice, remove the thermal plug (Figure 1) by loosening the four 1/4-inch slotted hex head machine screws, lift out the plug by the handle, and drop in the dry ice through the neck into the chamber below.
- d. To achieve the necessary high packing density, dry ice must be compacted by tamping as with the end of a wood two-by-four; using caution to prevent damage to instruments at the bottom of the dry ice chamber.
- e. Pack dry ice to fill the chamber as full as possible, with allowance for insertion of the thermal plug. Check that net weight is over 108 pounds before closing dry ice chamber.
- f. Reinstall thermal plug, and fasten securely to container.
- g. Recheck and record weights on Data Record for amount of dry ice in ice chamber on date of shipment.

11. Confirm that thermal plug is installed and secured.

12. Record pertinent information on data record of manual copy to be fastened inside instrument compartment.
Enclose spare manual and spare return label.

Install side panel and make sure it is securely fastened.

DIBORANE SHIPPING CONTAINERDATA RECORD - LOADINGPreliminary Checkout

1. Battery: ☐ Checked satisfactory (_____% on date_____)
☐ Replaced _____(date)

2. Insulation Space Pressure (vacuum)

Reading _____ microns @ _____ TI-12
_____ ambient

If repump is necessary:

Pump to _____ microns @ _____ TI-12
on _____ (date) _____ ambient

Checked _____ microns @ _____ TI-12
on _____ (date) _____ ambient

3. Pressure Test (_____ psig)

Leakage @ _____

Repair Completed _____ (date)

Precooling to -80°C. with Liquid Nitrogen

LN₂ Charged _____ on _____ (date)

LN₂ Charged _____ on _____ (date)

LN₂ Removed _____ on _____ (date)

Pressure test satisfactory on _____(date)

Diborane Charge

Analysis 100% N₂ on _____ (date)

Charged _____ lbs. B₂H₆ on _____ (date)

Topped to _____ lbs. B₂H₆ on _____ (date)

Charged _____ lbs. B₂H₆ on _____ (date)

Topped to _____ lbs. B₂H₆ on _____ (date)

Container Readings Immediately After Diborane Charge

PI-11 _____ psig Time _____

TI-12 _____ °C. Date _____

LI-13 _____ %

VI _____ %

Vacuum _____ Microns

Diborane Analysis _____ (if applicable).

Preparation for Shipment

1. ☐ HOV-1 Closed; two caps installed.
2. ☐ HOV-2 Closed; two caps installed.
3. ☐ HOV-14 Closed (gauge purge); cap installed.
4. ☐ HOV-15 Open (gauge shutoff valve).
5. ☐ Switch in "ON" position.
6. ☐ Tare Weight Stenciled on container.
7. ☐ "DOT SP 6522" Stenciled on container.
8. ☐ Container labeled according to DOT requirements (Manual Section VI).
9. ☐ Other labels installed as required (Manual Section VI recommendations).

10. ☐ Dry Ice Charged; 108 lbs. or more;
actual _____ lbs.
11. ☐ Thermal Plug Installed and Secured.
12. ☐ Manual copy (copies) in Instrument Com-
partment; secure side panel.

Container Readings Immediately Before Dry Ice Addition

PI-11 _____ psig Time _____

TI-12 _____ °C. Date _____

LI-13 _____ %

VI _____ %

Vacuum _____ Microns

LOADING DATA

Diborane - Gross _____ pounds

 Tare _____ pounds

 Net _____ pounds

Date Charged _____

Dry Ice - With Ice _____ pounds

 Without Ice _____ pounds

 Ice Weight _____ pounds

Date Iced for Shipment _____

APPROVED FOR SHIPMENT by _____
OPERATIONS SUPERVISOR

VI PROCEDURE FOR SHIPMENT

Diborane shipment in this container is controlled by Department of Transportation Special Permit Number 6522, a copy of which is in Section II. For shipment, diborane is classified as both "Compressed gas, n.o.s. (Flammable)" and "Poisonous gas, n.o.s.".

Those terms of DOT Special Permit 6522 pertinent to this manual section are as follows:

- A. Shippers of diborane must be authorized by registration of their identity with and receiving acknowledgement from the Department of Transportation, Hazardous Materials Regulation Board, Washington, D. C. 20590, and must have a copy of the special permit; these being required by Section 5 of DOT Special Permit No. 6522.
- B. According to Section 8 of DOT Special Permit No. 6522, the only authorized mode of transportation is motor vehicle. Furthermore, Section 9.e. requires that "Any common carrier by motor vehicle transporting diborane under the terms of this permit must be specifically approved by the Federal Highway Administration".
- C. A copy of DOT Special Permit No. 6522, kept current, must be carried aboard each motor vehicle, according to Section 9.a. of the permit.

- D. The container must have not less than 108 pounds of dry ice in the dry ice chamber on the date of shipment, according to section 7.a.ii. of DOT Special Permit 6522.
- E. Section 7.b. of DOT Special Permit No. 6522 places the following requirements on loading of the container with diborane.
1. Filling is to be by weight only.
 2. Container precooled to minus 80°C. with liquid nitrogen must reach destination within 15 days from date of shipment. True temperature at or below -80°C. must be confirmed by pressure of 18 psig or lower on date of shipment.
 3. Containers cooled to minus 70°C. with dry ice (or liquid nitrogen) must reach destination within the following schedule.
 - (a) 15 days from date of shipment when diborane net weight is 175 to 200 pounds.
 - (b) 12 days from date of shipment when diborane net weight is 100 to 174 pounds.True temperature at or below -70°C. must be confirmed by pressure of 32 psig or lower on date of shipment.
- F. Section 6 of DOT Special Permit No. 6522 requires that the quantity of diborane shipped be not less than 100 pounds* nor more than 200 pounds. Section 7.b. of the permit further requires that diborane quantity shall determine required delivery schedule of container cooled to -70°C., as defined

*Refer to Permit Section 6a (Page 27A) for special provisions to ship quantities less than 100 pounds.

by above item "E" of this manual section.

G. Sections 7.b.ii., 7.b.iii, and 9.b. of DOT Special Permit No. 6522 require that shipments of diborane reach destination within either 12 days or 15 days, depending on quantity of diborane and initial temperature, as defined by above item "E" of this manual section.

H. Container labeling required by DOT is as follows:*

1. DOT Special Permit 6522 Section 7.a.i. requires that:

"In addition to the flammable gas label and the poison gas label, each outside shipping container must bear a conspicuous label reading as follows:

'IF NOT DELIVERED BEFORE _____ CARRIER
MUST ADVISE (Insert name and address of shipper), ALSO
THE BUREAU OF EXPLOSIVES, WASHINGTON, D.C. BY WIRE.'

The date inserted in the blank space on this label must not be in excess of the number of days prescribed herein from the date shipment is offered for transportation."

2. DOT Special Permit 6522 reference 49 CFR 171.6 requires that "DOT SP 6522" be plainly and durably marked on portable tanks in letters at least 2 inches high on a contrasting background.

* See NOTE at the end of this section for labeling which is not required by DOT, but which should be used to prevent mishandling of container and to properly identify the contents.

J. Because of the requirement for delivery at destination within 12 or 15 days (as defined by above item "E" of this manual section), the following terms of the permit (Sections 9.c. and 9.d.) will also apply.

1. "Each shipping paper must show thereon, following the commodity description, the notation, appropriately executed:

DOT SPECIAL PERMIT NO. 6522
DATE OF SHIPMENT _____
IF NOT DELIVERED BEFORE _____
DAYS CARRIER MUST ADVISE BUREAU
OF EXPLOSIVES, WASHINGTON, D.C.
BY WIRE."

2. "Each shipper must require acknowledgement of receipt of shipment from consignee by wire, to be confirmed in writing, and must promptly notify the Bureau of Explosives (AAR) of any such shipment not received at destination within two days after shipment is due."

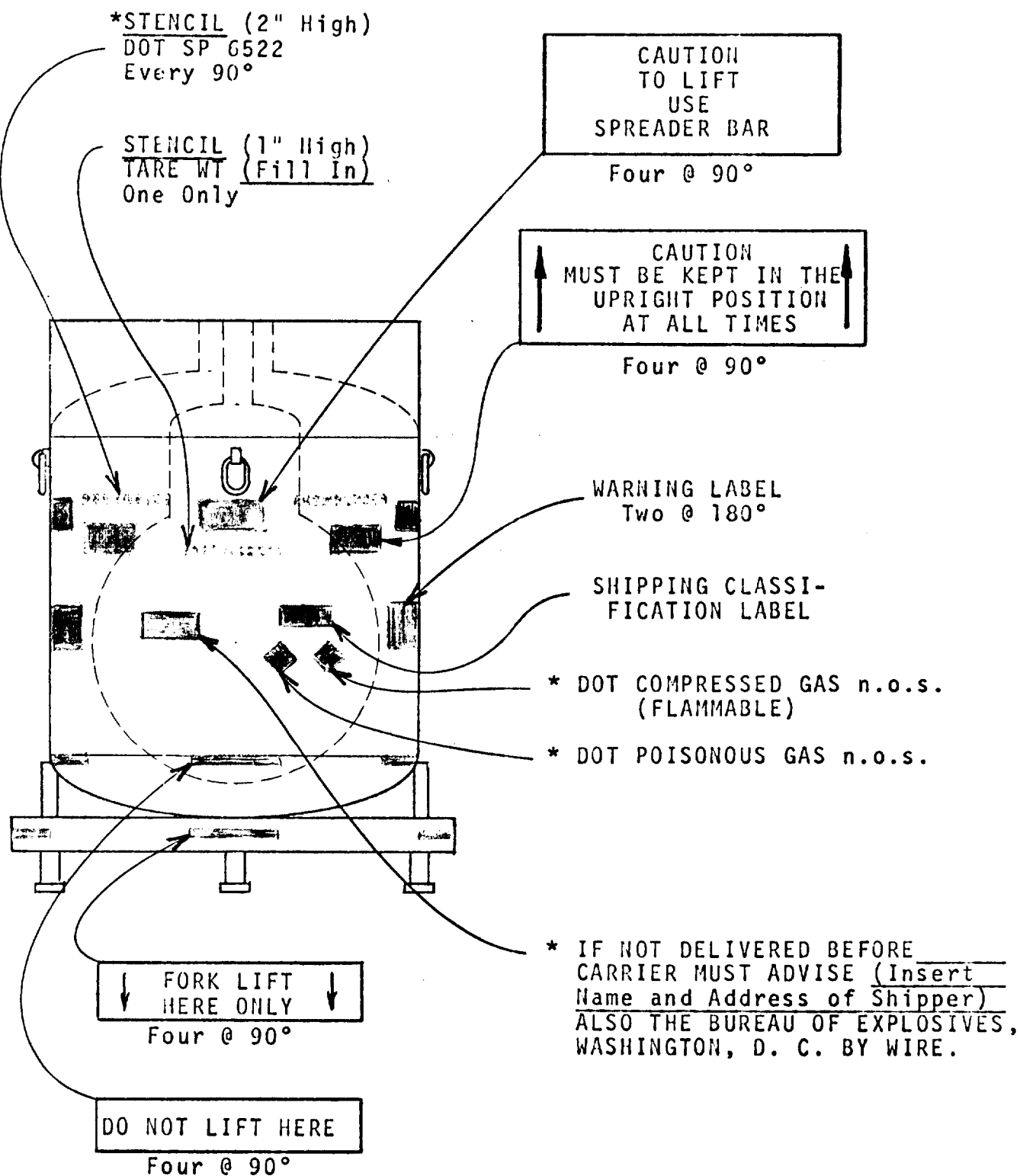
CAUTION: The above items summarize some of the more important requirements specified in DOT Special Permit 6522; however, THIS SHOULD NOT BE INTERPRETED AS A COMPLETE SET OF INSTRUCTIONS, which of necessity would incorporate all of the DOT regulations except those specifically waived by the special permit. RESPONSIBILITY FOR COMPLIANCE WITH ALL APPLICABLE REGULATIONS RESTS WITH THE SHIPPER.

NOTE: The following labels are not required by DOT, but should be used to prevent mishandling of the container and to identify contents. Figure 24 shows recommended location of these labels.

1. "Fork Lift Here Only", on 3-3/4" X 13-1/4" label with arrow (down) on both sides, to show where to position forks. Four required.
2. "Do Not Lift Here", on 4" X 14" label, to show where not to position forks. Four required.
3. "Caution: To Lift Use Spreader Bar", on 7-1/4" X 13" label, to prevent damage to top enclosure. Four required.
4. "Caution: Must Be Kept In The Upright Position At All Times", on 7" X 14" label, to warn against turning the container on its side. Four required.
5. Warning label containing suitable precautionary information on potential hazards, first aid, fire-fighting, etc.; 7-3/4" X 7". Two required.
6. Shipping classification label, identifying "Compressed Gas, n.o.s. (Flammable)" and "Poisonous Gas, n.o.s.", as well as "DOT Special Permit 6522"; 5" X 8". One required.

FIGURE 24

DIBORANE SHIPPING CONTAINER
RECOMMENDED LABELING



* Required by DOT

DIBORANE SHIPPING CONTAINERDATA RECORD - SHIPPING

From Loading Data Record - Diborane Charge

Gross Weight _____ lbs. (no dry ice)

Tare Weight _____ lbs.

Net Diborane _____ lbs.

From Loading Data Record - Dry Ice Charge

Shipping Weight _____ lbs.

Above Gross _____ lbs.

Net Dry Ice _____ lbs.

Check List

1. ☐ Shipper has acknowledgement from DOT Hazardous Materials Regulation Board as authorized shipper.
2. ☐ Motor vehicle carrier approved by Federal Highway Administration for transport of diborane, in accordance with Section VI.B. of this manual.
3. ☐ Shipping papers prepared in accordance with Section VI.G.1. of this manual.
4. ☐ Copy of DOT Special Permit No. 6522 (current) available for carrier.
5. ☐ Container properly labeled, in accordance with Section VI.F. of this manual.

6. ☐ Diborane net weight between 100 and 200 pounds
(actual _____ lbs.).
7. ☐ Dry Ice net weight at least 108 pounds (actual _____
lbs.) on shipping date (_____).
8. ☐ Operating Manual copy (copies) included inside instru-
ment compartment.

APPROVED FOR SHIPMENT by _____
SHIPPER

VII PROCEDURE FOR RECEIVING

A. Unloading Container from Truck

The container may be moved either by fork lift truck or by crane. The following precautions should be noted:

1. Fork lift truck
 - a. Use a fork lift truck with capacity for at least 3000 pounds at a distance of 30 inches. Fork length must be at least 32 inches, and spread of forks should be at least 30 inches outside-to-outside.
 - b. Follow all standard safety practices for use of fork lift trucks. Be sure to block both front and rear wheels of truck from which container is removed before driving fork lift onto bed of truck.
 - c. The weight coming off the truck will be 2900 pounds plus the weight of the fork lift truck. Suitable supports or tiedowns may be necessary to prevent tip up of the truck.

2. Crane

- a. Use a crane with capacity for at least 3000 pounds at the distance the container will be handled. Use a spreader bar to separate drop chains at least 50 inches; drop chains should be at least 30 inches long.
- b. Follow all standard safety practices for use of cranes.

B. Action Required on Date Received

1. Check Pressure

Immediately after receiving the container, pressure should be checked to verify a safe condition. Pressure may be checked by reading the gauge (PI-11) through the plexiglass window, without removing the side panel. The gauge shutoff valve (HOV-15) is left open* during shipment. Record pressure on data sheet in manual copy fastened to the container inside the instrument compartment.

Normally the pressure will be about 60 psig for a container delivered in 15 days (for 200 pounds initially at -80°C.). Pressure will rise about 15 psi per day between the 15th day and the 20th day.

*At the earliest opportunity, remove side panel and confirm that valve is open (so that the gauge does in fact read the actual container pressure).

2. Check for Leakage

Immediately after delivery, the container should be checked for leaks with a suitable borane detector. Customer may use the same practice employed with the smaller cylinders containing smaller quantities of diborane. Also inspect piping for white solids deposits as evidence of slight leakage.

If leakage is detected, appropriate action must be taken immediately. Using the necessary personnel protective equipment, it may be necessary to move the container to a more suitable area. Then depending on the source and magnitude of the leak, either repair or immediate transfer may be dictated.

3. If Necessary, Add Dry Ice

It would be desirable to arrange unloading of the container as soon as possible after delivery; and in that case it would not normally be necessary to add dry ice prior to unloading. This would permit accurate weighing without interference caused by changes in dry ice weight.

It is recommended that dry ice be added when pressure reaches 100 to 150 psig. The dry ice should be broken into small lumps, either with an ice crush-

ing machine or manually. Initially dry ice usage will be about 15 pounds per day.

To add dry ice, remove the thermal plug (Figure 1) by loosening the four 1/4-inch slotted hex head machine screws, lift out the plug by the handle, and drop the dry ice through the neck into the chamber below. To achieve a higher packing density, dry ice may be compacted by tamping with the end of a wood two-by-four; using caution to prevent damage to instruments at the bottom of the dry ice chamber.

4. Acknowledge receipt of shipment to shipper
 - a. by wire and
 - b. confirm in writing.

NOTE: This is required by Section 9d. of DOT Special Permit 6522.

5. Record other data and pertinent information on data sheet in manual copy that remains with the shipping container (see next page).

DIBORANE SHIPPING CONTAINERDATA RECORD - RECEIVING

Received @ _____ (time) on _____ (date).

Pressure Reading _____ psig

Remarks: _____

Check of Gauge Shutoff Valve:

Result _____

Check for Leakage:

(1) Borane Detector

Result _____

(2) Visual Inspection

Result _____

Note whether dry ice was added (when and how much) _____

Receipt of shipment acknowledged to shipper on _____ (date)

CONTAINER RECEIVED SATISFACTORY by _____
OPERATIONS SUPERVISOR

VIII PROCEDURE FOR STORAGE

A. Short Term Storage (to about 5 days after receipt, or 20 days after shipment of 200 pounds initially at $-80^{\circ}\text{C}.$).

1. For short term storage of a few days, it may not be necessary to add dry ice before unloading the diborane (See "Procedure for Receiving", Section VII.B.3.).
2. Test alarm (See "Container Design, Instruments and Controls", Section I.C.5.) to assure that it is operable.
3. Record pressure and temperature daily, using the data sheet in the manual copy fastened to the container inside the instrument compartment.
 - a. If no dry ice is added, pressure should rise at about 15 psi per day between the 15th day and the 20th day (see Table 3, Page 24).
 - b. If dry ice is added, pressure should drop to about 30 to 40 psig and stabilize around 30 psig as long as adequate dry ice is kept in the chamber (see Figure 16, Page 26).
 - c. Any highly abnormal rate of pressure rise should be reported to supervisory personnel for evaluation and recommendations.

- B. Long Term Storage (longer than about 5 days after receipt, or 20 days after shipment of 200 pounds initially at $-80^{\circ}\text{C}.$).

1. Record Data.

Container pressure and temperature should be recorded daily to assure that no unsafe condition has developed. Weekend readings could be omitted if it creates inconvenience. Use the data sheet in the Manual copy fastened to the container inside the instrument compartment.

Report any unusual readings or other observations to a supervisor.

2. Maintain Dry Ice Supply.

For extended storage, dry ice should be added immediately upon receipt of the container; and the supply would be replenished as required thereafter.

It is recommended that dry ice be added whenever the pressure begins to rise, which will be detected by daily readings. This will keep pressure about 30 to 40 psig and temperature about -65 to $-70^{\circ}\text{C}.$, at which diborane decomposition is negligible. Decomposition should not be a problem below $-35^{\circ}\text{C}.$, which corresponds to about 150 psig.

For convenience two 50-pound cakes of dry ice can be added at a time, after breaking them into small lumps either manually or with an ice crushing machine. Initially dry ice consumption will be about 15 pounds per day, and this should drop to about 10 pounds per day as long as an ice supply is maintained.

To add dry ice, remove the thermal plug (Figure 1) by loosening the four 1/4-inch slotted hex head machine screws, lift out the plug by the handle, and drop the dry ice through the neck into the chamber below. To achieve a higher packing density, dry ice may be compacted by tamping with the end of a wood two-by-four; using caution to prevent damage to instruments at the bottom of the dry ice chamber.

3. Leave Side Panel In Place.

The cold carbon dioxide (CO₂) gas vented from the dry ice chamber is piped to the valve quadrant to provide some cooling in that area, where diborane vapors up to the valves and gauge are exposed to ambient temperature. Keeping the side panel on to close this compartment will provide some cooling of that area to reduce decomposition rate. It will also prevent loss of the side panel.

4. Test Alarm.

It is recommended that the alarm be tested (See Container Design, Instruments and Controls", Section I.C.5.) once a week to assure it remains operable.

IX PROCEDURE FOR UNLOADING

WARNING: DIBORANE SAFETY AND HANDLING INFORMATION IN SECTION III OF THIS MANUAL MUST BE READ AND UNDERSTOOD BEFORE PROCEEDING WITH UNLOADING OF DIBORANE.

Detailed procedures for unloading of diborane into a storage facility of necessity depend upon the design of that facility; therefore, these instructions can be complete only in terms of the shipping container itself. The customer must prepare additional procedures for the facility receiving the diborane.

Section 4 of DIBORANE HANDBOOK (N70-78384) by M. T. Constantine et al. of the Research Division of Rocketdyne (a Division of North American Rockwell Corporation) prepared for NASA under Contract NAS7-769 contains very complete information on Storage and Handling of diborane. All applicable sections of that compilation should be considered in both facility design and worker education/training.

The "Safety and Handling Procedures" Section 5.4.2 of DIBORANE HANDBOOK, included in Section V of this manual, are also applicable to this section on Procedure for Unloading.

General Transfer Procedures

(From DIBORANE HANDBOOK, Section 4.5.3)

4.5.3 Transfer of B₂H₆ From Shipping Container

Diborane can be discharged from its storage container either by its own vapor or gas pressure, or by pressurizing the container with dry nitrogen or helium. Inert gas pressurization is used almost exclusively at the present time for unloading large amounts of liquid B₂H₆, because this technique is extremely reliable. Vapor pressure unloading is used primarily in the transfer of small quantities of liquid or gaseous B₂H₆.

The transfer system must be chemically compatible with B₂H₆, leak-proof, and in excellent operating order. The complete system must also be completely passivated, dried, and purged with dry nitrogen or helium gas prior to the commencement of complete flow conditions. It should be maintained free of air, moisture, and other contamination.

In preparing for a transfer operation, all personnel not directly concerned with the operation shall evacuate the hazard area. Appropriate warning lights and signs shall be displayed to keep out unauthorized personnel. Personnel performing the transfer operation shall wear the fully protective equipment described in Section 6.4.1.2. If the operations are performed remotely, at least two operating personnel should be fully dressed to facilitate proper spill and fire control. Sufficient safety equipment should be available for all personnel allowed to remain in the hazard area. Supervisory and emergency support personnel shall be notified prior to executing any hazardous operation in the storage area.

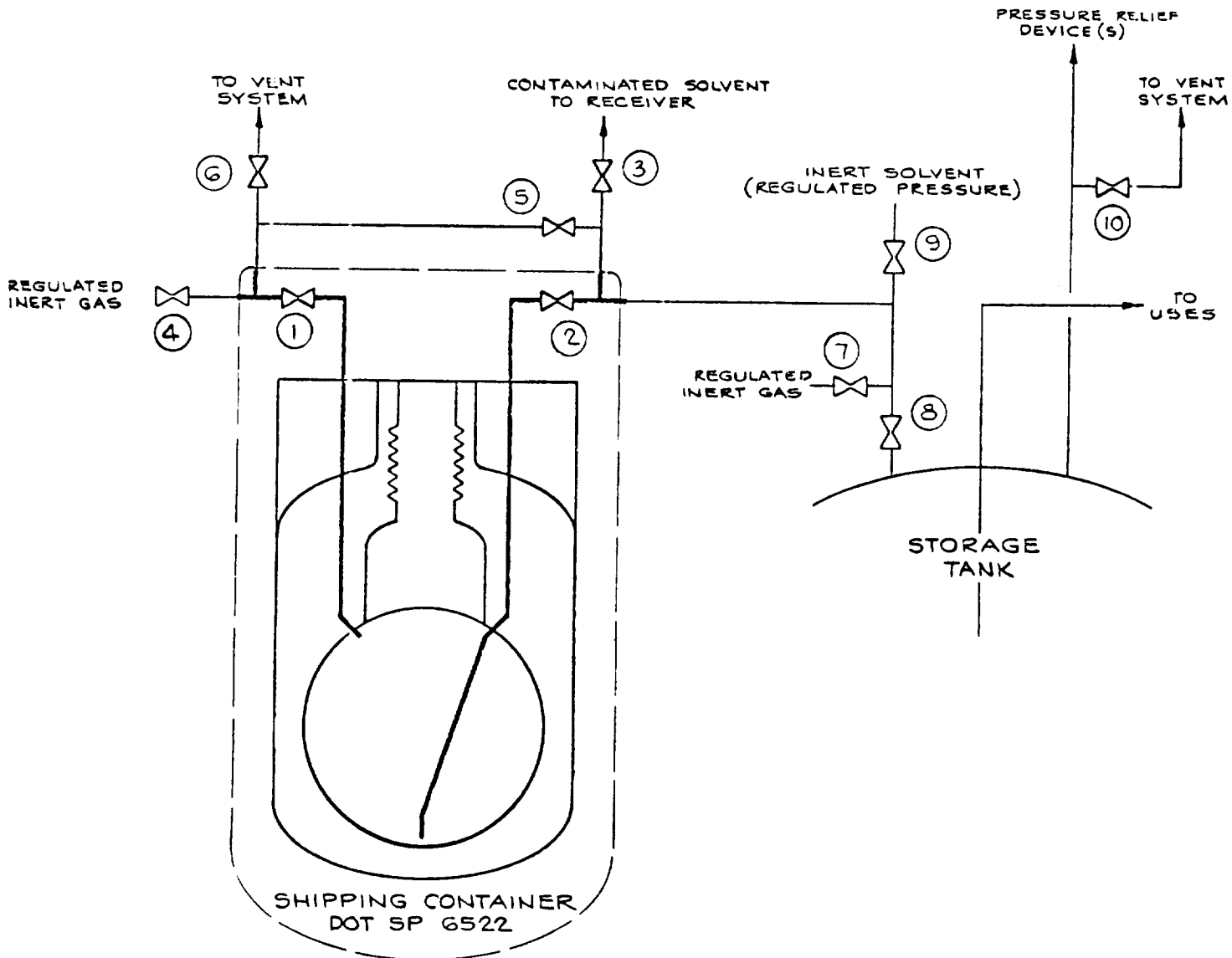
UNLOADING

A. Transfer of Liquid By Inert Gas Pressurization

Callery's experience is based entirely on the use of dry nitrogen as the inert gas for pressurization. Dry helium is the inert gas preferred by some users (because of high nitrogen gas solubility in diborane); and Callery knows of no reason to not use dry helium. Carbon dioxide should not be used.

For use of either inert gas, the procedure is given below; valve numbers refer to Figure 25, a simplified schematic diagram of what is presumed to be a typical unloading facility:

1. This procedure presumes that the transfer lines are clean, and therefore it is only necessary to purge lines to displace the air which may be present if not totally sealed under inert gas blanket. Furthermore this procedure presumes that the entire system has been properly maintained and tested to be free of any leaks. THE OPERATOR MUST NOT PRESUME, HE MUST KNOW THAT THE SYSTEM IS CLEAN AND FREE OF LEAKS.
2. All valve shown in Figure 25 are to be closed at the start.

SIMPLIFIED SCHEMATIC DIAGRAM
CONTAINER UNLOADING PIPING

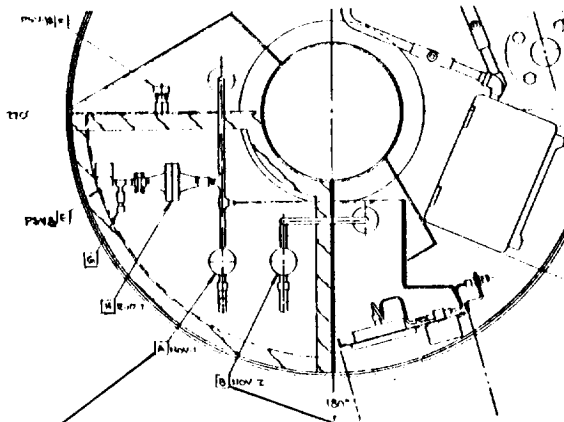
- (1) Container Valve - Gas pressurization
- (2) Container Valve - Liquid Outlet
- (3) Solvent to waste receiver
- (4) Pressurizing Gas Supply
- (5) Container Bypass Valve
- (6) Container and Transfer Line Vent
- (7) Purge Gas Supply
- (8) Storage Tank Inlet Shutoff
- (9) Inert Solvent Supply
- (10) Storage Tank Vent

3. Position and secure the container.
4. Remove side panel and set aside for reassembly after transfer has been completed. This exposes about half of the circumference of the container down about one foot from the top, providing access to both the valve quadrant and the instrument quadrant.
5. It is desirable, but not necessary, to connect the downstream side of the container pressure relief valve into an emergency vent header. Thus if the rupture disk and relief valve were opened by overpressure, the diborane could be piped to a more remote point of release. Such piping should be of an inside diameter at least as large as 3/4-inch Schedule 40 pipe, and should contain a minimum number of elbows (use 45-degree instead of 90-degree elbows). This line should contain NO manual shutoff valves, and should discharge open to the atmosphere. The relief valve outlet is 3/4-inch female NPT, so it will accept a 3/4-inch male NPT connection. Flexible connection should be made to prevent placing any strain on the relief valve and/or its inlet piping.

6. Identify the two valves where connections are to be made: (Check that both valves are closed).

Figure 26

Unloading Valve Locations



Note that pressurization valve (1) is on the left, and its tubing line also connects to the pressure relief devices. The liquid outlet valve (2) is on the right, nearer to the partition between the valve quadrant and the instrument quadrant.

HOV-1 (See also Figure 2 and Figure 3)
= 1 Figure 25
Top of Tank
Pressurization
or Vent

HOV-2 (See also Figure 2 and Figure 3)
= 2 Figure 25
Dip Tube
Liquid Outlet

7. Carefully remove caps from tees where connections are to be made. In each connection, just crack the fitting loose first; then proceed to loosen and remove unless observations dictate otherwise. Keep caps for use on the container return shipment.
8. Connect lines from storage facility to shipping container. These lines should be flexible to prevent placing any strain on container valves and piping. MAKE ABSOLUTELY

SURE LINES ARE CONNECTED TO THE CORRECT FITTING (see item 6 above). These lines may be connected loosely for initial purging.

9. Purge transfer line by flowing inert gas through, as follows:
 - a. Open valves (4) and (6), and purge for 3 to 5 minutes*; then close valves (6) and (4).
 - b. Open valves (7) and (3), and purge for 3 to 5 minutes*; then close valve (3), and leave valve (7) open.
 - c. Open valves (5) and (6), and purge for 3 to 5 minutes*. Loosen the connections to the tees at valve (1) and valve (2) during this purge; then tighten all four fittings securely.
 - d. Close valve (6), and leave valve (5) and valve (7) open. This will permit pressure buildup to a predetermined level for leak testing of the connections and transfer line.
10. When system is shown to be free of any leaks, proceed to final purging by pressure and release; according to the following steps:

*Purging time required depends upon many factors which differ in individual systems (such as line diameter, line length, purge rate available, previous use of line, etc.). The diborane user is warned to be sure purge time is more than adequate.

- a. Close valve (7), and open valve (6) to vent; then close valve (6).
 - b. Repeat two more times: open valve (7) until pressure builds up; then close valve (7) and open valve (6) until vented; then close valve (6).
 - c. Close valve (5). All system valves should now be closed again, with system containing dry inert gas.
11. Set inert gas pressure regulator to control desired pressure on container for unloading, depending on desired unloading rate.
- a. Measured unloading rates in tests using liquid diborane at -38°C . were as follows:
 - 0.23 lb/sec (4.6 GPM) @ $\Delta P = 20$ psi
 - 0.30 lb/sec (6.0 GPM) @ $\Delta P = 35$ psi
 - b. Inert gas pressure 10 to 50 psi higher than pressure in the shipping container should cover most applications. This differential should never be less than 10 psi, and maximum container operating pressure should not exceed 400 psig.
12. Open valve (4), admitting inert gas up to valve on container; then open container valve (1) to pressurize inner tank of container. Watch container pressure gauge (PI-11) for pressure to stabilize at desired setting.

13. Open container valve (2) to open diborane to transfer line; then open valve (8) from transfer line into storage tank.
14. Continue until all of the diborane (or the desired amount) has been transferred.
 - a. When the container goes "empty" (that is, when the liquid seal is lost), inert gas will escape through the container dip tube to the storage tank; and container pressure will drop (unless inert gas is fed through a very high capacity regulator).
 - b. Depending on the facility and the desire to minimize the amount of inert gas fed to the storage tank, it may be helpful to use one or more instruments (such as flowmeter in transfer line, scale for container, or level indicator on storage tank) to detect the end of the transfer. The container level indicator may also be useful for this purpose.
15. When the liquid seal is lost, close valve (2) to stop inert gas flow into storage tank. VALVE (8) MUST BE LEFT OPEN.
16. When container pressure has built up again, open valve (2) to confirm that container is "empty" (gas instead of liquid is transferred); then close valve (4) inert

gas supply and container valve (1). Leave valve (2) open until container pressure vents to between 25 and 50 psig, then close container valve (2).

17. Container is now closed, with inert gas pressure of 25 to 50 psig; and system is ready for purging.
18. Open inert gas purge valve (4) and container bypass valve (5), and purge transfer line into storage tank for about 5 minutes*. It may be necessary to vent the storage tank to maintain flow of the inert gas purge (this procedure cannot be specific in terms of a storage system whose design is unknown).
19. Close inert gas purge supply valve (4), bypass valve (5), and storage tank inlet valve (8). Immediately open the transfer line to a vent, either via valve (6) to the vent system or via valve (3) to a vented solvent receiver. This will protect against the circumstance where some liquid may have been heldup in the line.

NOTE: Procedure can be varied at this point. This procedure will be written for use of an inert solvent such as kerosene (water-free). A thorough purging with inert gas might be substituted if properly planned and executed.

*Purging time required depends upon many factors which differ in individual systems (such as line diameter, line length, purge rate available, previous use of line, etc.). The diborane user is warned to be sure purge time is more than adequate.

20. If valve (6) had been opened to vent system, close valve (6) and open valve (3) to vented receiver for contaminated solvent. Open inert dry solvent supply valve (9) to purge solvent through transfer line for 3 to 5 minutes*.
21. Close solvent supply valve (9), and open inert gas purge valve (7) to purge solvent from line into receiver for 3 to 5 minutes*.
22. Close valve (7) and valve (3).
23. Open inert gas supply valve (4) and open vent valve (6) to purge vent line for 3 to 5 minutes*; then close valve (6) and valve (4).
24. Carefully disconnect lines from shipping container, just cracking loose each fitting first before loosening and removing.
25. Replace the four caps on the two tees on the shipping container valves (1) and (2), and tighten.
26. Disconnect piping that may have been connected to container relief valve (after any necessary purging, depending where this had been connected into).
27. Record activity on shipping container data sheet

*Purging time required depends upon many factors which differ in individual systems (such as line diameter, line length, purge rate available, previous use of line, etc.). The diborane user is warned to be sure purge time is more than adequate.

28. Side panel may be installed at this time if container is to be moved, or may be left off until preparing container for return.
29. If container is weighed to confirm less than 5 pounds of diborane, be sure side panel is also on scale and that any necessary corrections are made for dry ice in the ice chamber. Container tare weight will be stenciled on container and recorded on manual copy with the container.
30. Notify all personnel concerned that the transfer operation is completed.
31. Procedures for disposal of contaminated solvent are beyond the scope of this manual. The user should have developed this and other facilities/procedures in advance, using the previously referenced DIBORANE HANDBOOK where necessary for guidance.
32. Arrange for return of container, according to Section X following.

B. Other Types of Transfer

1. Transfer of liquid by diborane vapor pressure (instead of inert gas pressurization).
 - a. This method of unloading is not recommended because of the difficulty in controlling pressure. There is no convenient way to heat the container,

so this method of transfer could involve a long time period.

- b. If it should become necessary or desirable to remove all or part of the diborane by vapor pressure, the same procedure applies except that the application of inert gas pressure to the container is omitted.

2. Transfer of diborane gas

- a. Transfer of all the diborane as a gas would be impractical for the reasons outlined above, and is not recommended for that purpose.
- b. To remove a small amount of diborane, this procedure might be used; but details of the procedure would depend on the system into which the diborane is transferred. The user is cautioned to use proper procedures for purging before and after.

DIBORANE SHIPPING CONTAINERDATA RECORD - UNLOADING

Method: ☐ Remove liquid by inert gas pressurization.
☐ Remove liquid by vapor pressure.
☐ Remove gas.

Data:

Inert gas used _____

Pressure _____ psig (maximum)

Storage Tank Pressure @ start _____ psig

@ finish _____ psig

Container Readings: Start* Finish**

Pressure, Psig _____ _____

Temperature, °C. _____ _____

Level, % _____ _____

Voltage, % _____ _____

* Before Pressurization

** After Venting

Transfer Time _____

Date (s) Unloaded _____

Remarks:

READY FOR RETURN-UNLOADING APPROVED by _____
OPERATIONS SUPERVISOR

X PROCEDURE FOR RETURN

RETURN SHIPMENTS OF "EMPTY" CONTAINERS MUST COMPLY WITH THE TERMS OF DOT SPECIAL PERMIT 6522.

- A. To return the empty container, the customer must register their identity with and receive acknowledgement from the Department of Transportation, Hazardous Materials Regulation Board; Washington, D. C. 20590 to become an authorized shipper in accordance with Section 5 of DOT Special Permit 6522.
- B. For return shipment the container must be verified to be "empty", as defined by Section 7.c.i. of DOT Special Permit 6522.
 - 1. The empty weight must not exceed the marked tare weight by more than five pounds; or
 - 2. Level of liquid diborane must be below the bottom of the dip tube; that is, only vapor is vented from the liquid opening. Loss of liquid seal will be evident by ability to vent gas pressure from the container (to user's tank or other proper vent system) through the dip tube. (See Section IX, "Procedure for Unloading").

3. Pressure should be vented to between 25 and 50 psig at the time container is emptied. Refer to Section IX of this manual for procedures to purge and disconnect lines used for unloading diborane.
 4. For return shipment of containers which do not qualify as "empty", refer to Section XI of this manual.
- C. The container must have not less than 108 pounds of dry ice in the dry ice chamber on the date of return shipment, according to Section 7.a.ii. of DOT Special Permit 6522.
- D. Section 7.c.iii. of DOT Special Permit 6522 requires that containers shipped as empty reach destination within 15 days from date of shipment (see also labeling, below).
- E. Labeling of the empty container for return is as follows:
1. DOT Special Permit 6522 Section 7.a.i. requires that:
"In addition to the flammable gas label and the poison gas label, each outside shipping container must bear a conspicuous label reading as follows:
'IF NOT DELIVERED BEFORE _____ CARRIER MUST ADVISE
(Insert name and address of shipper), ALSO THE BUREAU
OF EXPLOSIVES, WASHINGTON, D.C., BY WIRE.' The date
inserted in the blank space on this label must not be
in excess of the number of days prescribed herein from
the date shipment is offered for transportation."

2. One blank of the above label ("If not delivered before _____ carrier must...") should be enclosed with the manual fastened to the container, for convenience of the customer to use on the return shipment. The customer is required to confirm that all other necessary labels remain intact and legible for the return shipment, or to replace these as necessary.

3. DOT Special Permit 6522 reference 49 CFR 171.6 requires that "DOT SP 6522" be plainly and durably marked on portable tanks in letters at least 2 inches high on a contrasting background. Since this will have been stenciled on for shipment of the full container, it should only be necessary to confirm that this marking is still clearly legible.

F. Because of the requirement for delivery at destination within 15 days (DOT Special Permit 6522, Section 7.c.iii.), the following terms of the permit (Section 9.c. and 9.d.) will also apply.

1. "Each shipping paper must show thereon, following the commodity description, the notation, appropriately executed:

DOT SPECIAL PERMIT NO. 6522
DATE OF SHIPMENT _____
IF NOT DELIVERED BEFORE _____
DAYS CARRIER MUST ADVISE BUREAU
OF EXPLOSIVES, WASHINGTON, D.C.
BY WIRE."

2. "Each shipper must require acknowledgement of receipt of shipment from consignee by wire, to be confirmed in writing, and must promptly notify the Bureau of Explosives (AAR) of any such shipment not received at destination within two days after shipment is due."
- G. According to Section 8 of DOT Special Permit 6522, the only authorized mode of transportation is motor vehicle. Furthermore, Section 9.e. requires that "Any common carrier by motor vehicle transporting diborane under the terms of this permit must be specifically approved by the Federal Highway Administration".
- H. A copy of DOT Special Permit 6522, kept current, must be carried aboard each motor vehicle, according to Section 9.a. of the permit.

CAUTION: The above items summarize some of the more important requirements specified in DOT Special Permit 6522; however, THIS SHOULD NOT BE INTERPRETED AS A COMPLETE SET OF INSTRUCTIONS, which of necessity would incorporate all of the DOT regulations except those specifically waived by the special permit. RESPONSIBILITY FOR COMPLIANCE WITH ALL APPLICABLE REGULATIONS RESTS WITH THE SHIPPER; that is, the customer for return shipments.

DIBORANE SHIPPING CONTAINER

DATA RECORD - RETURN

1. ☐ Container emptied by removing liquid through the dip tube until liquid seal is lost (gas is vented to user's storage tank through dip tube).
 - *2. ☐ Container gross weight (without dry ice) not more than five (5) pounds higher than original tare weight (stenciled on container, and recorded on DATA RECORD for both Loading and Shipping).
 3. ☐ Container vented to between 25 psig and 50 psig.
Actual Pressure _____psig. Temperature _____°C.
Date _____
 4. ☐ Pressurizing valve (1) and dip tube valve (2) both closed. Gauge shutoff valve (15) open. Caps installed on all connections.
 5. ☐ At least 108 pounds of dry ice in ice chamber on date of shipment _____. Actual quantity of dry ice _____pounds. Thermal plug fastened.
 6. ☐ Container properly labeled, in accordance with Section X.E. of this manual.
 7. ☐ Copy of DOT Special Permit No. 6522 available for carrier.
-

*This may be omitted only if item 1 is completed to assure that the container is "empty".

8. ☐ Shipping papers prepared in accordance with Sections X.F.1. and X.G. of this manual.
9. ☐ Upon completion of above, place this copy of manual inside valve compartment; and attach side panel securely.

APPROVED FOR RETURN SHIPMENT by _____
OPERATIONS SUPERVISOR

XI PROCEDURE FOR RECOOLING & RESHIPMENT

- A. Before attempting to follow this procedure, the reader should understand pertinent information in Section I (Design and Performance) of this manual. In particular, see "Item B., Recooling" under "Performance" on page 23, and Figure 16 on page 26.
- B. Shipment of a recooled container may be necessary for basically two reasons:
1. Containers are sometimes delayed in transit to the point where neither delivery nor return can be accomplished within the required time.
 2. Insufficient precooling and/or charging of diborane at too high a temperature may prevent achieving a pressure below 32 psig, required to initiate a shipment.
- In either above case, shipment may be made after the necessary recooling with dry ice.
- C. Dry ice addition may be accomplished through the following procedure:
1. Breakup the dry ice into small lumps, either with an ice crushing machine or manually.

2. To add dry ice, remove the thermal plug (Figure 1) by loosening the four 1/4-inch slotted hex head machine screws, lift out the plug by the handle, and drop in the dry ice through the neck into the chamber below.
3. To achieve the necessary high packing density, dry ice must be compacted by tamping as with the end of a wood two-by-four; using caution to prevent damage to instruments at the bottom of the dry ice chamber.
4. Pack dry ice to fill the chamber reasonably full, with allowance for insertion of the thermal plug.
 - a. In the case of a container delayed in transit to or beyond the 12- or 15-day limit, it is important to add even a small quantity of dry ice quickly, providing time that may be necessary to secure larger quantities.
 - b. On the last charge of dry ice just prior to shipment (or reshipment) it is necessary that the ice chamber be filled as completely as possible to obtain the required 108 pounds of dry ice.
5. Reinstall thermal plug, and fasten securely to container.

- D. Recooling for either reason MUST be continued through repeated dry ice charges until inner tank pressure (PI-11) is down to at least 32 psig. In the example shown in Figure 16 (page 26), three dry ice charges totaling 234 pounds brought pressure down to 32 psig; additional dry ice would have been required to bring the total charge to 108 pounds before making a shipment.
- E. Refer to Section VI (Procedure for Shipment) for other information on other requirements of DOT Special Permit No. 6522 for shipment of diborane in this container.
- F. Shipments containing less than 100 pounds of diborane may also be re-cooled and reshipped.
1. Above Sections XI. A. through XI. D. inclusive are applicable.
 2. All of the requirements of DOT S.P. 6522 Section 6a (page 27A) remain in effect.

DIBORANE SHIPPING CONTAINER

DATA RECORD - RECOOLINGINITIAL CONDITION:

Time _____ PI-11 _____ psig
Date _____ TI-12 _____ °C.

Dry Ice (visual observation whether any in dry ice chamber).

ACTION TAKEN:

<u>Time</u>	<u>Date</u>	<u>Dry Ice Added</u>	<u>PI-11 psig</u>	<u>TI-12 °C.</u>
_____	_____	_____	_____	_____
_____	_____	_____	_____	_____
_____	_____	_____	_____	_____
_____	_____	_____	_____	_____
_____	_____	_____	_____	_____
_____	_____	_____	_____	_____
_____	_____	_____	_____	_____
_____	_____	_____	_____	_____
_____	_____	_____	_____	_____

FINAL CONDITION:

Shipping Date _____

PI-11 _____ psig TI-12 _____ °C.

Dry Ice _____